

# Toxicological Assessment and Strategic Risk Mitigation of Heavy Metal Contamination in Shampoos: A Case Study from Aden, Yemen.

**Abstract:** The safety and toxicological profile of cosmetic products have gained significant global attention due to potential health risks arising from heavy metal contamination. This study provides a comprehensive toxicological assessment of key heavy metals—Arsenic (As), Nickel (Ni), Cobalt (Co), and Copper (Cu)—in twelve popular shampoo brands (coded S1 to S12) marketed in Aden, Yemen.

**Methodology** involved quantitative analysis using high-sensitivity Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) following standardized wet acid digestion, with rigorous Quality Assurance (QA) maintained using analytical reagent-grade chemicals and high-purity water ( $18.0 \text{ M}\Omega\cdot\text{cm}^{-1}$ ). Statistical validation via **One-way ANOVA** and **LSD tests** ( $p \leq 0.05$ ) revealed alarming systemic non-compliance with international safety standard. Specifically, **Cobalt** concentrations in **66.6%** of the samples exceeded the WHO permissible limit, while **Nickel** levels in **91.6%** of the investigated brands surpassed the US permissible threshold of **0.6  $\mu\text{g/g}$** . **Arsenic** contamination was found to be critical in **33.3%** of the samples, with concentrations reaching a peak of **45.2  $\mu\text{g/g}$** , significantly exceeding the WHO limit of **10.0  $\mu\text{g/g}$** ; however, **100%** of the samples remained within safe limits for **Copper**. These findings indicate that continuous dermal exposure facilitates the bioaccumulation of toxic metal ions, which form stable, hazardous complexes with cellular protein functional groups (**-COOH**, **-NH<sub>2</sub>**, and **-SH**), posing grave long-term risks including systemic toxicity and potential carcinogenicity. Beyond mere detection, this study proposes a **Strategic Risk Mitigation Framework**, that integrates mandatory manufacturing oversight and specialized port screening with a consumer transition toward validated herbal alternatives as a safer public health roadmap.

**Keywords:** Aden-Yemen, Bioaccumulation, Heavy Metals, ICP-OES, Shampoo ,Strategic Mitigation, Toxicological Assessment

## 1. INTRODUCTION

Over the past few decades, the safety of cosmetic products and their constituent ingredients has attracted increasing global attention, leading to the evolution of toxicological safety determination as a specialized discipline since the latter half of the 20<sup>th</sup> century [Safavi 2019]. The cosmetic industry has experienced a significant boost, producing a vast array of products for skin, hair, and body care—including creams, beauty soaps, and shampoos—that have become integral to daily hygiene [Chauhan et al., 2010]. The global cosmetic industry is experiencing rapid expansion, with revenues projected to exceed USD 800 billion by 2030. However, this growth is accompanied by significant concerns regarding the toxicological and environmental impacts of synthetic ingredients, which contribute to pollution and systemic toxicity [Rachmawati et al., 2025]. However, growing scientific evidence has identified the presence of toxic elements across various personal care categories [Mahmoodabad et al., 2019; Lim et al., 2018]. Mercury, cadmium, lead, nickel, chromium, and copper are among the most frequently detected heavy metals in products such as shampoos, lipsticks, and powders [Volpe et al., 2012]. Significant concerns have been raised regarding specific chemical ingredients found in shampoos, including halogenated organic compounds, formaldehyde, musk fragrances, and heavy metals, which are potentially linked to carcinogenic risks [Onna et al., 2021]. While heavy metals like lead, arsenic, cadmium, and mercury are known to cause chronic systemic effects, others such as **chromium, nickel, and cobalt** act primarily as **skin sensitizers**. The accumulation of these elements within the human body is associated with a spectrum of chronic conditions, including reproductive disorders, neurological impairments, hair loss, and various malignancies [Feizi et al., 2019]. Among these, arsenic (As), lead (Pb), cadmium (Cd), mercury (Hg), nickel (Ni), and chromium (Cr) are most commonly implicated in **human toxicity** [Kishan et al., 2014]. These heavy metals occur naturally in the environment—sourced from soil, rocks, and water—and are often inadvertently introduced into cosmetics through contaminated raw materials used during manufacturing. In the absence of comprehensive legal frameworks regulating toxic levels in many regions, the "**Threshold of Toxicological Concern**" (TTC) has been proposed as a vital conceptual benchmark for evaluating ingredient safety [Yashim et al., 2000; Kroes et al.,

2007]. Furthermore, the toxicological risk posed by these products is multifaceted, depending on the metal concentration, the product's chemical synthesis, dermal penetration capacity, the total surface area of exposure, and the frequency of use [Feizi et al.,2019; Mahmood et al, 2022]. The underlying mechanism of toxicity involves the absorption of heavy metal ions into the human body, where they form stable complexes with the **carboxylic acids (-COOH)**, **amines (-NH<sub>2</sub>)**, and **thiols (-SH)** of proteins. This biochemical interaction leads to cellular damage, potential cell death, and various systemic diseases. Management of metal intoxication typically requires the use of chelating agents that bind with metal ions to form complexes for removal from the body [Flora and Pachauri, 2010; Zainy, 2017; Saleh et al., 2020].

In the specific context of Yemen, there is growing concern regarding the safety of inexpensive shampoos sold widely in the Aden governorate. Many of these products are imported from countries characterized by weak regulatory oversight, substandard manufacturing conditions, and a lack of rigorous screening. Research regarding the contamination of shampoos with toxic metals in Yemen remains extremely rare, and there is a notable deficiency in local control and consumer awareness regarding the levels of hazardous metals in these products. The toxicological landscape of cosmetics in Yemen has recently come under intense scrutiny. Over the last decade, analytical investigations have consistently revealed high risks of heavy metal exposure across different product categories in Aden. Initial studies highlighted significant contamination in skin-whitening creams [Saleh et al., 2016], followed by very recent findings of extreme Lead (Pb) levels in lipsticks and foundation creams—reaching up to 331.75 ppm—documented in 2025 [Fadhel, Saleh, & Binabood, 2025]. This current research aims to extend this toxicological surveillance to the hair care sector, specifically targeting widely-used shampoo brands.

Research regarding the contamination of shampoos with toxic metal's in Yemen remains extremely rare, and there is a notable deficiency in local control. Recent toxicological reviews emphasize that the transition from laboratory detection to effective regulation of heavy metals like Nickel and Chromium is crucial for mitigating public health risks, as these metals remain among the most prevalent skin sensitizers in modern cosmetics [Adam-Dima et al., 2024]. Consequently, this study was designed to bridge the existing gap in local

regulatory oversight. It aims not only to conduct a **toxicological assessment** of key heavy metals—specifically Arsenic (As), Nickel (Ni), Cobalt (Co), and Copper (Cu)—using high-sensitivity ICP-OES but also to develop a **strategic risk mitigation roadmap** for **public health safety in Aden**. By synthesizing experimental data with a proposed strategic framework, **this research addresses the biochemical risks of bioaccumulation and provides actionable solutions for both regulators and consumers in the region.**

## **2. MATERIALS and METHODS**

### **2.1. Sampling and Sample Selection**

A total of twelve [Flora and Pachauri, 2010] commercially available shampoo brands were selected for the quantitative determination of heavy metals (As, Ni, Co, and Cu). These brands represent the most popular choices among consumers in the local markets of Aden, Yemen. The samples included products imported from various countries, namely the United Arab Emirates (UAE), China, Saudi Arabia, and France. Detailed information regarding the sample codes, country of origin, and intended use is summarized in **Table 1**.

To ensure the reliability of the data, three replicates of each sample were prepared and analyzed prior to their respective expiration dates, following the systematic research methodology illustrated in **Figure 1**.

### **2.2. pH Determination**

The pH of the shampoo samples was measured using a digital pH meter equipped with a glass electrode, following the standard analytical protocols established by **APHA (1995)**. For each measurement, 5 g of the sample was homogenized with 20 mL of deionized water in a glass beaker. The mixture was then processed in an ultrasound device for 20 minutes to ensure complete dispersion and homogeneity before recording the pH values.

**Table 1. Identification Codes, Manufacturing Origin, and Intended Use of the Analyzed Samples**

| Sample No. | Sample ID | Country of Origin | Intended Use |
|------------|-----------|-------------------|--------------|
| 1          | S1        | UAE               | Children     |
| 2          | S2        | China             | Children     |
| 3          | S3        | Saudi Arabia      | Antidandruff |
| 4          | S4        | Saudi Arabia      | Died hair    |
| 5          | S5        | Saudi Arabia      | Antidandruff |

|    |     |              |                 |
|----|-----|--------------|-----------------|
| 6  | S6  | Saudi Arabia | Strengthen hair |
| 7  | S7  | Saudi Arabia | Dry hair        |
| 8  | S8  | Saudi Arabia | Normal hair     |
| 9  | S9  | Saudi Arabia | Destroyed hair  |
| 10 | S10 | Saudi Arabia | Strengthen hair |
| 11 | S11 | France       | Normal hair     |
| 12 | S12 | China        | Hair care       |

### 2.3. Reagents and Analytical Standards

All chemical reagents utilized in this study were of analytical reagent grade. All solutions were prepared using Millipore-purified water with a conductivity exceeding  $18.0 \text{ M}\Omega\cdot\text{cm}^{-1}$ . Concentrated Nitric acid ( $\text{HNO}_3$ ) and Perchloric acid ( $\text{HClO}_4$ ) were sourced from BDH (England) for the sample digestion process. Standard multi-element solutions for Arsenic (As), Nickel (Ni), Cobalt (Co), and Copper (Cu) were prepared via serial dilution from 1000 ppm certified stock solutions.

### 2.4. Sample Preparation and Acid Digestion

To facilitate the quantification of heavy metals, the organic matrix of the shampoo was decomposed using a wet acid digestion procedure as described in validated literature [Ojezele et al., 2018]. Specifically, 1 g of each sample was accurately weighed and digested in 5 mL of a concentrated acid mixture ( $\text{HNO}_3\text{:HClO}_4$  at a 3:1 ratio). The mixture was heated on a hot plate at a controlled low temperature for 2–3 hours until the appearance of white vapors, signaling the completion of the digestion. The resulting digestates were then diluted to a final volume of 10 mL with triple-distilled water and filtered through Whatman No. 1 filter paper to obtain a clear solution for subsequent metal quantification.

### 2.5. Instrumental Analysis

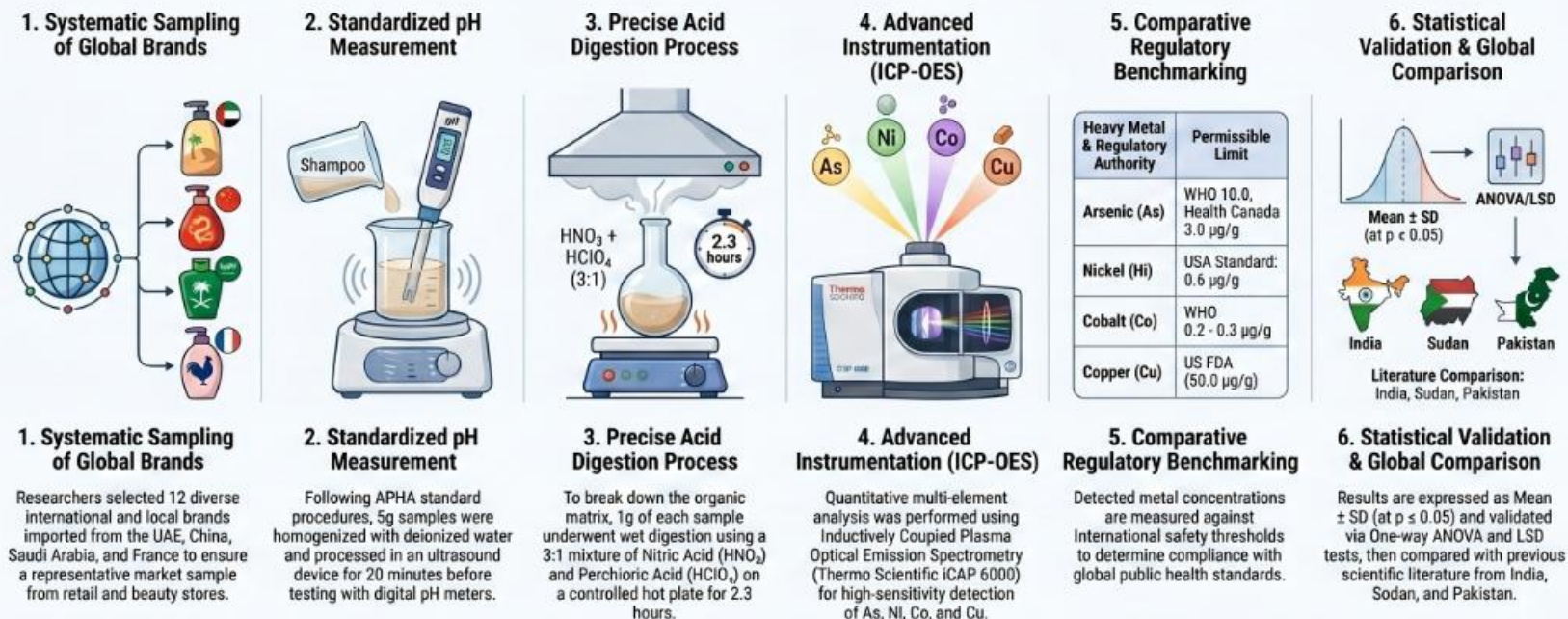
The concentrations of Arsenic, Nickel, Cobalt, and Copper were determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES, Thermo Scientific iCAP 6000 series, North America). This advanced instrumentation provided high-sensitivity multi-element detection. Each sample was analyzed in triplicate to ensure precision, and the final concentrations were expressed as the mean value  $\pm$  SD (standard deviation).

### 2.6. Quality Control and Quality Assurance (QC/QA)

The instrumentation (ICP-OES Thermo Scientific iCAP 6000) was calibrated using certified multi-element stock solutions (1000 ppm), from which working standards were prepared via precise serial dilutions [Saleh, 2021]. To monitor potential contamination during the wet acid digestion process ( $\text{HNO}_3\text{:HClO}_4$ , 3:1 ratio), reagent blanks were prepared and analyzed in parallel with the shampoo samples [Saleh et al., 2016]. Furthermore, all samples were analyzed in triplicate ( $N=3$ ) to assess instrumental precision, and results were reported as the **Mean  $\pm$  Standard Deviation (SD)** to provide a clear measure of analytical consistency [Saleh, 2021; Islam et al., 2015].

## 2.7. Statistical Analysis

The experimental data were subjected to comprehensive statistical evaluation to determine the significance of the results. Statistical processing was performed using Origin 7.5 software (OriginLab Corporation, Northampton, MA, USA), which is a robust platform for analytical chemistry data validation. This methodology aligns with previously validated protocols for shampoo analysis in the region [Saleh, 2021]. A One-way Analysis of Variance (**ANOVA**) based on a Completely Randomized Design (**CRD**) was employed to assess whether the differences in heavy metal concentrations between the twelve shampoo brands were statistically significant. For post-hoc comparisons between the means of different brands, the **Least Significant Difference (LSD)** test was applied at a confidence level of  $p \leq 0.05$  [Saleh, 2021; Islam et al., 2015]. In the reported **Table 2**, the presence of different alphabetical superscripts (**a, b, c**) indicates significant differences between the means, while identical letters signify no statistical difference, thereby ensuring a transparent and rigorous interpretation of the toxicological data [Saleh, 2021; Al-Bassam and Taka, 2011].



**Figure 1: Systematic Analytical Methodology for the Quantitative Determination of Toxic Heavy Metals in Shampoo Samples**

**Figure 1.** Systematic Analytical Methodology for the Quantitative Determination of Toxic Heavy Metals in Shampoo Samples. The workflow integrates six rigorous stages: (1) Systematic sampling of 12 global brands from UAE, China, Saudi Arabia, and France; (2) Standardized pH measurement using APHA protocols; (3) Precise wet acid digestion (3:1  $\text{HNO}_3$ : $\text{HClO}_4$ ); (4) High-sensitivity ICP-OES instrumental analysis; (5) Comparative regulatory benchmarking against WHO, Health Canada, and the USA Standard (0.6  $\mu\text{g/g}$  for Ni); and (6) Robust statistical validation via One-way ANOVA and LSD tests at  $p \leq 0.05$ .

### 3. RESULTS and DISCUSSION

#### 3.1. pH Values of Shampoo Samples

The pH levels of the investigated shampoo brands ranged from 5.113 to 6.706, with the lowest value recorded for the S11 sample and the highest for S4 (**Table 2**). Statistical analysis (at  $p \leq 0.05$ ) indicated significant differences between samples, likely due to variations in formulations and manufacturing standards. When compared to the standards set by the central authority for standardization and quality (4.6–8.0 for adults; 4.6–7.0 for children) [Oyedeji et al., 2011], all samples fell within the permissible range, except for the S1 and S2 samples which were notably close to the lower limit. The overall pH profile, as visualized in **Table 2**, suggests that most brands maintain a safe physiological range for scalp application.

#### 3.2. Heavy Metals Concentrations and Associated Toxicological Risks

"The overall safety and regulatory compliance profile of the investigated shampoo samples is summarized in Table 2 and Figure 2. While the samples demonstrated a high degree of safety regarding **Copper (Cu)** levels (100% pass rate against the US FDA limit of **50 µg/g**) and maintained a safe physiological **pH** range between **5.1** and **6.7**, alarming violations were observed for other toxic elements.

Specifically, toxicological benchmarking of **Arsenic (As)** concentrations revealed that **33.3%** of the samples exceeded the **WHO** limit of **10.0 µg/g**. Furthermore, a more stringent assessment showed that **50%** of the brands surpassed the **Health Canada** threshold of **3.0 µg/g**, reaching a critical peak of **45.2 µg/g** in sample **S10** (Saudi Arabia).

**Table 2.** Quantitative Assessment of pH and Heavy Metal Concentrations (µg/g) in Coded Shampoo Samples (Mean ± SD, N=3)

| Sample No. | Sample ID | pH                           | As                             | Co                             | Cu                       | Ni                            |
|------------|-----------|------------------------------|--------------------------------|--------------------------------|--------------------------|-------------------------------|
| 1          | S1        | 6.430 <sup>a</sup> ±0.0152   | 0.2 <sup>af</sup> ±0.001       | <b>0.8<sup>ai</sup>±0.003</b>  | 0.4 <sup>ah</sup> ±0.004 | BDL                           |
| 2          | S2        | 5.953 <sup>bn</sup> ±0.0252  | BDL                            | <b>1.7<sup>b</sup>±0.004</b>   | 0.3 <sup>bj</sup> ±0.003 | <b>1.0<sup>a</sup>±0.004</b>  |
| 3          | S3        | 5.173 <sup>ck</sup> ±0.0152  | <b>45.0<sup>bh</sup>±0.098</b> | <b>1.1<sup>cei</sup>±0.014</b> | 20.5 <sup>c</sup> ±0.058 | <b>1.9<sup>b</sup>±0.013</b>  |
| 4          | S4        | 6.706 <sup>de</sup> ±0.0208  | <b>17.3<sup>c</sup>±0.215</b>  | <b>1.9<sup>d</sup>±0.010</b>   | 0.5 <sup>de</sup> ±0.002 | <b>15.6<sup>c</sup>±0.032</b> |
| 5          | S5        | 6.666 <sup>efj</sup> ±0.0208 | 2.8 <sup>d</sup> ±0.021        | <b>1.2<sup>ehi</sup>±0.011</b> | 0.5 <sup>eg</sup> ±0.006 | <b>24.8<sup>d</sup>±0.043</b> |
| 6          | S6        | 6.613 <sup>fj</sup> ±0.0152  | <b>5.5<sup>e</sup>±0.028</b>   | <b>0.4<sup>f</sup>±0.005</b>   | 0.6 <sup>f</sup> ±0.012  | <b>14.4<sup>e</sup>±0.035</b> |
| 7          | S7        | 5.876 <sup>gh</sup> ±0.0151  | 0.2 <sup>f</sup> ±0.006        | BDL                            | 0.5 <sup>g</sup> ±0.005  | <b>17.1<sup>f</sup>±0.032</b> |
| 8          | S8        | 5.933 <sup>h</sup> ±0.0153   | BDL                            | <b>0.3<sup>g</sup>±0.003</b>   | 0.4 <sup>h</sup> ±0.005  | <b>19.9<sup>g</sup>±0.076</b> |

|       |             |                            |                          |                          |                         |                          |
|-------|-------------|----------------------------|--------------------------|--------------------------|-------------------------|--------------------------|
| 9     | S9          | 5.263 <sup>i</sup> ±0.0057 | 11.1 <sup>g</sup> ±0.043 | 0.5 <sup>hi</sup> ±0.002 | 0.8 <sup>i</sup> ±0.002 | 12.5 <sup>h</sup> ±0.03  |
| 10    | S10         | 6.613 <sup>j</sup> ±0.0305 | 45.2 <sup>h</sup> ±0.223 | BDL                      | 0.3 <sup>j</sup> ±0.003 | 12.8 <sup>i</sup> ±0.072 |
| 11    | S11         | 5.113 <sup>k</sup> ±0.0252 | 6.5 <sup>j</sup> ±0.046  | 0.8 <sup>i</sup> ±0.008  | 0.9 <sup>k</sup> ±0.002 | 13.2 <sup>j</sup> ±0.161 |
| 12    | S12         | 6.403 <sup>l</sup> ±0.0310 | BDL                      | BDL                      | 1.2 <sup>l</sup> ±0.002 | 9.8 <sup>k</sup> ±0.044  |
| Range | Min.        | 5.113                      | BDL                      | BDL                      | 0.3                     | BDL                      |
|       | Max.        | 6.706                      | 45.2                     | 1.9                      | 20.5                    | 24.8                     |
| L.S.D | L.S.D       | 0.1005                     | 0.420                    | 0.4266                   | 0.0815                  | 0.2850                   |
| MPL   | WHO         |                            | 10                       | 0.2-0.3                  |                         |                          |
|       | Canada      |                            | 3.0                      |                          |                         |                          |
|       | US FDA      |                            |                          | 5                        | 50                      |                          |
|       | US Standard |                            |                          |                          |                         | 0.6                      |

**Note:** Different alphabetical superscripts (a, b, c...) within the same column indicate statistically significant differences ( $p \leq 0.05$ ) between samples according to the LSD test.

BDL: Below detection limit; SD: Standard Deviation; LDS: Least Significant Difference; MPL: Maximum Permissible Limits.

### 3.2.1. Arsenic (As) Concentrations and Toxicological Risks

Toxicological benchmarking of **Arsenic (As)** levels against international safety thresholds showed that **33.3%** of the analyzed products exceeded the WHO permissible limit of 10.0 µg/g [ALqadami et al., 2013; Saleh et al., 2016]. Furthermore, **50% of the samples surpassed the more stringent Health Canada threshold of 3.0 µg/g** [Chauhan et al., 2014; Saleh et al., 2016]. These elevated concentrations, reaching a **peak of 45.2 µg/g in sample S10 (Table 2)**, present a serious public health risk regarding chronic dermal exposure. As visualized in the compliance profile (**Figure 2**). Furthermore, the concentrations found in this study are substantially higher than previous regional baselines, a comparison detailed in **Figure 2** and **Table 3**.

Statistical analysis using One-way ANOVA and LSD tests  $p \leq 0.05$  confirmed that the high-contamination group, specifically S10 and S3 (45.0 µg/g), exhibited arsenic levels significantly higher than all other brands and previous local baselines in Aden [Saleh et al., 2016]. These detected levels are substantially more alarming than those reported in global literature, including studies from India (0.42 µg/g) [Islam et al., 2015].

From a public health perspective, chronic dermal exposure to such elevated concentrations of arsenic is highly concerning, as it is a potent systemic toxin and carcinogen. Long-term accumulation is clinically linked to **hyperpigmentation, hyperkeratosis, and the development of basal and squamous cell carcinomas** [Gibb et al., 2001; Sun et al., 2014; Bhattacharjee et al., 2013; Zeng & Zhang, 2020; Sun et al., 2009].

The **high bioaccumulation risk** observed in samples like S10 and S3 necessitates **urgent regulatory intervention**. The concentration of arsenic in the investigated samples followed this decreasing order: **S10 (45.2) > S3 (45.0) > S4 (17.3) > S9 (11.1) > S11 (6.5) > S6 (5.5) > S5 (2.8) > S1 (0.2) = S7 (0.2) > S2, S8, S12 (BDL)**.

### 3.2.2. Nickel (Ni) Concentrations and Associated Toxicological Risks

Nickel concentrations in the investigated brands ranged from BDL to a **peak of 24.8 µg/g**, with the highest value observed in **sample S5 (Saudi Arabia) (Table 2)**. Alarming statistical results showed that **91.6% of the samples (11 out of 12 brands) surpassed the US permissible threshold of 0.6 µg/g** [Omenka & Adeyi, 2016; Umar & Caleb, 2013]. This extensive violation of the US safe limit is a critical finding, as visualized in the compliance summary (**Figure 2**), and underscores the urgent necessity for mandatory routine screening of imported cosmetic products to mitigate the risks of allergic contact dermatitis and potential carcinogenicity. The high levels of Nickel found in this study corroborate recent findings by **Adam-Dima et al. (2024)**, who highlighted the persistent challenge of managing Nickel contamination in personal care products and its direct correlation with the rising incidence of allergic contact dermatitis globally. These findings pose a significant risk for severe contact dermatitis and potential carcinogenicity, as metallic nickel is classified as possibly carcinogenic to humans [Health Canada, 2007-2009; Sahu et al., 2014; Gorel et al., 1997; IARC, 2010; Sainio et al., 2000]. Clinical evidence suggests that concentrations as low as 1 ppm can trigger pre-existing allergies, with the first cosmetic-induced nickel allergy documented over a decade ago [Sainio et al., 2000].

Statistical Analysis of Nickel (Ni) Concentrations: Statistical evaluation of the experimental data was performed using Origin 7.5 software, employing a One-way ANOVA under a Completely Randomized Design (**CRD**). To distinguish significant variations between the twelve coded shampoo brands, the Least Significant Difference (**LSD**) test was applied at a significance level of **p ≤ 0.05**. All results are presented as the **Mean ± Standard Deviation (SD)** of **triplicate** measurements to ensure high analytical precision.

The analysis revealed that Nickel concentrations ranged from Below Detection Limit (BDL) to a peak of **24.8 µg/g** recorded in sample **S5 (Saudi Arabia)**, while sample S1 (UAE) was the only brand that remained below detection limits (**Table 2**). The LSD test confirmed statistically significant differences ( $p \leq 0.05$ .) between the investigated samples, indicating a high degree of variability in manufacturing quality and raw material purity across different brands. The nickel concentrations in the investigated samples in decreasing order were:

**S5 (24.8) > S8 (19.9) > S7 (17.1) > S4 (15.6) > S6 (14.4) > S11 (13.2) > S10 (12.8) > S9 (12.5) > S12 (9.8) > S3 (1.9) > S2 (1.0) > S1 (BDL).**

**S5 (Saudi Arabia) > S8 (Saudi Arabia) > S7 (Saudi Arabia) > S4 (Saudi Arabia) > S6 (Saudi Arabia) > S11 (France) > S10 (Saudi Arabia) > S9 (Saudi Arabia) > S12 (China) > S3 (Saudi Arabia) > S2 (China) > S1 (UAE).**

### 3.2.3. Cobalt (Co) Concentrations and Safety Assessment

Cobalt (Co) was detected in **75%** of the investigated shampoo samples (**Table 2**), with concentrations ranging from Below Detection Limit (BDL) to a maximum of **1.9 µg/g** recorded in **sample S4 (Saudi Arabia)**. A critical toxicological evaluation revealed that **66.6% of the samples exceeded the WHO permissible limit of 0.2–0.3 µg/g** [Ekere et al., 2014; Saleh et al., 2016], highlighting a significant lack of compliance with international safety standards for these products. Despite these violations, all samples remained within the US FDA permissible limit of 5.0 µg/g [Saleh et al., 2016; Sukender et al., 2012], as shown in the comparative data in **Table 3**.

Statistical validation via One-way ANOVA demonstrated significant variations among the samples, identifying S4 and S2 (1.7 µg/g) as the most contaminated brands. From a toxicological standpoint, cobalt is a recognized **potent skin sensitizer and allergen** [Fischer et al., 2017; Iwegbue, et al., 2016]; its presence in all samples raises concerns regarding chronic allergic contact dermatitis for frequent users [Iwegbue et al., 2017; Samia et al., 2017]. The concentration of cobalt in the studied samples followed this decreasing order: **S4 (1.9) > S2 (1.7) > S5 (1.2) > S3 (1.1) > S1 (0.8) = S11 (0.8) > S9 (0.5) > S6 (0.4) > S8 (0.3) > S7, S10, S12 (BDL).**

### 3.2.4. Copper (Cu) Levels and Compliance

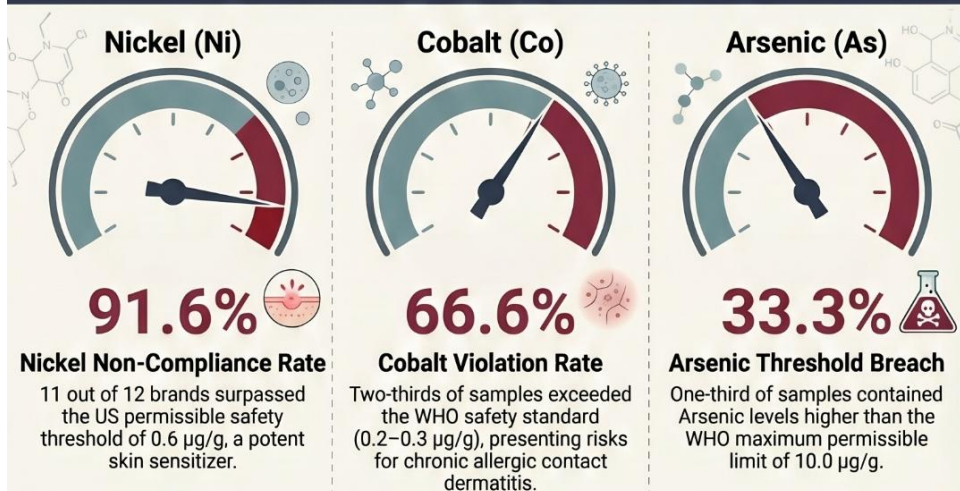
The concentration of Copper (Cu) in the sampled shampoos, as presented in **Table 2**, ranged from 0.3 to 20.5 µg/g, with the peak concentration found in sample S3 (Saudi Arabia). In contrast to the other heavy metals, 100% of the investigated brands were found to be safe regarding copper content, as all levels remained well below the US FDA permissible limit of 50.0 µg/g [Saleh et al., 2016; Sukender et al., 2012] (**Figure 2**). While copper is an essential trace element for hemoglobin synthesis, its presence in cosmetics as an impurity from substandard raw materials can still pose a risk of localized skin irritation in hypersensitive individuals [Fischer et al., 2017]. The quantitative results showed that S3 had a significantly higher copper profile compared to other brands ( $p \leq 0.05$ ). The copper concentrations in decreasing order were:

**S3 (20.5) > S12 (1.2) > S11 (0.9) > S9 (0.8) > S6 (0.6) > S4 = S5 = S7 (0.5) > S1 = S8 (0.4) > S2 = S10 (0.3).**

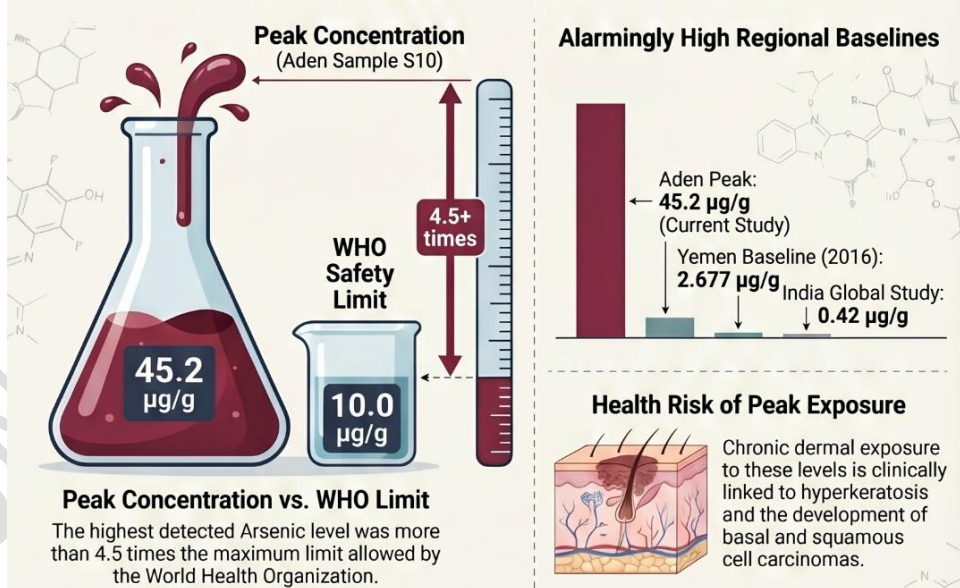
# Toxicological Non-Compliance and Heavy Metal Benchmarking in Commercial Shampoos

Analysis of twelve popular shampoo brands sold in Aden, Yemen, using high-sensitivity ICP-OES reveals systemic failure to meet international safety standards for heavy metals, posing severe long-term health risks.

## Global Safety Non-Compliance Rates



## Peak Toxicity Benchmarking: Arsenic (As)



**Statistical Validation:** Confidence Level:  $p \leq 0.05$ .  
All variations in heavy metal concentrations were statistically significant.  
**Analytical Framework:** ANOVA and LSD tests ensured rigorous interpretation.

**Figure 2: Comparative Toxicological Profile and Quantitative Compliance Analysis**

### 3.3. Comparison of the results of this study with previous studies

The comparative data presented in **Table 3** and **Figure 2**, highlights that the levels of Arsenic, Cobalt, and Nickel in this study are generally higher than those reported in earlier literature from India and Pakistan [Ullah et al., 2017; Islam et al., 2015]. This suggests a deteriorating trend in the quality of imported shampoos in the Aden market.

To contextualize these findings within a global and regional framework, **Figure 2** illustrates a comparative analysis of maximum metal concentrations. The data reveals, that Arsenic levels in the current study (**45.2 µg/g**) are significantly higher than those recorded in previous synthetic shampoo studies in India (0.42 µg/g) [Islam et al., 2015] and local 2016 baselines in Yemen (2.677 µg/g) [Saleh et al., 2016]. Furthermore, the figure correlates these elevated concentrations with specific health risks, such as hyperkeratosis and squamous cell carcinomas for Arsenic, and severe allergic contact dermatitis for Nickel and Cobalt exposure. This alarming trend of heavy metal contamination in the Aden market is further reinforced by very recent toxicological assessments of facial cosmetics, which documented extreme Lead (Pb) levels in lipsticks and foundation creams reaching as high as 331.75 ppm and 234.75 ppm, respectively [Fadhel, Saleh, & Binabood, 2025]. Taken together, these studies—spanning from initial reports on skin-whitening creams [Saleh et al., 2016] to the current findings in shampoos and makeup—confirm that consumers in Yemen are facing a cumulative and multifaceted chemical threat across a wide range of daily-use personal care products.

**Table 3.** Comparative Assessment of Heavy Metal Ranges in Studied Samples against Regional Studies and International Standards

| Country | As                          | Co | Cu | Ni | Ref. |
|---------|-----------------------------|----|----|----|------|
|         | Concentration (range; µg/g) |    |    |    |      |

|                           |                 |                     |                  |                     |                                                                |
|---------------------------|-----------------|---------------------|------------------|---------------------|----------------------------------------------------------------|
| India (Synthetic Shampoo) | 0.003-0.42      | -                   | 0.06-1.02        | 0.05- <b>5.9</b>    | Islam et al., 2015                                             |
| India (Herbal Shampoo)    | 0.001-0.02      | -                   | 0.1-0.41         | BLD-0.05            | Islam et al., 2015                                             |
| Sudan                     | 1.504-6.796     | -                   | -                | -                   | Sabah et al., 2013                                             |
| Pakistan                  | -               | 0.183- <b>1.225</b> | 0.02- <b>302</b> | 0.095- <b>1.610</b> | Ullah et al., 2017                                             |
| Yemen                     | 1.02-2.677      | 0.2-0.3             | 2.779-3.634      | 0.472- <b>1.426</b> | Saleh et al., 2016                                             |
| <b>Yemen</b>              | <b>BLD-45.2</b> | <b>BLD-1.9</b>      | 3-20.5           | <b>BLD-24.8</b>     | <b>This present study</b>                                      |
| WHO Limits                | <b>10.0</b>     | <b>0.2-0.3</b>      | -                | -                   | Saleh et al., 2016; ALqadami et al. 2013                       |
| Health Canada Limits      | <b>3.0</b>      | -                   | -                | -                   | Chauhan et al., 2014; Saleh et al. 2016                        |
| US FDA Limits             | -               | <b>5.0</b>          | <b>50.0</b>      | -                   | Saleh et al., 2016; Sukender et al., 2012; Chauhan et al. 2014 |
| USA standard              | -               | -                   | -                | <b>0.6</b>          | Umar&Caleb, 2013; Omenka & Adeyi, 2016                         |

BLD: Below detection limit

### 3.4. Strategic Framework for Mitigation (Figure 3)

#### Connecting Metal Concentrations to Biochemical Risks

The broader implications of the experimental results and their associated health risks are synthesized into a comprehensive roadmap for risk management, as illustrated in the Strategic Framework for Public Health Safety and Mitigation (**Figure 3**) [Adam-Dima et al., 2024; Safavi, 2019]. This framework addresses the critical nexus between biochemical toxicity, systemic regulation, and consumer safety through three integrated pillars [Mahmood et al., 2022].

#### 3.4.1. Bioaccumulation Pathophysiology: The Danger Process

The framework highlights the long-term dangers of **bioaccumulation pathophysiology**, where toxic heavy metal ions (As, Ni, and Co) are absorbed through the scalp via dermal penetration. Once these ions enter the biological system, they form stable, hazardous complexes by binding with the functional groups of cellular proteins—specifically **carboxylic acids (-COOH)**, **amines (-NH<sub>2</sub>)**, and **thiols (-SH)** [Flora & Pachauri, 2010; Zainy, 2017]. As depicted in **Figure 3**, this biochemical interaction leads to **protein denaturation** and

progressive cellular damage [Flora & Pachauri, 2010; Zainy, 2017]. This cumulative process is the primary mechanism behind the chronic systemic toxicity, reproductive impairments, and carcinogenic disorders linked to the high metal concentrations detected in this study [Safavi, 2019; Feizi et al., 2019].

### 3.4.2. Regulatory Action Plan: The Systemic Solution

To address the systemic non-compliance identified—most notably the **66.6%** violation rate for **Cobalt**, **33.3%** for **Arsenic** and **91.6%** for **Nickel**—a dual-action regulatory approach is essential [Adam-Dima et al., 2024]. The urgency of this intervention is further justified by cumulative toxicological evidence in the Aden market, where hazardous metal contamination has been consistently documented across various categories—ranging from skin-whitening creams (Saleh et al., 2016) and facial cosmetics, including lipsticks and foundation reaching Lead levels of 331.75 ppm (Fadhel, Saleh, & Binabood, 2025), to the synthetic shampoos analyzed in this study.

1. **Mandatory Manufacturing Controls:** Rigorous monitoring of raw materials and production phases is required to prevent contamination before products reach the market [Ojezele et al., 2018].
2. **Specialized Port Laboratories:** The establishment of dedicated testing facilities at ports of entry in Aden is critical. These laboratories must conduct routine analytical screening of all imported shampoo brands to ensure strict adherence to the safety thresholds established by the **WHO**, **US FDA**, and **Health Canada** [Mahmood et al., 2022; Saleh, 2021].

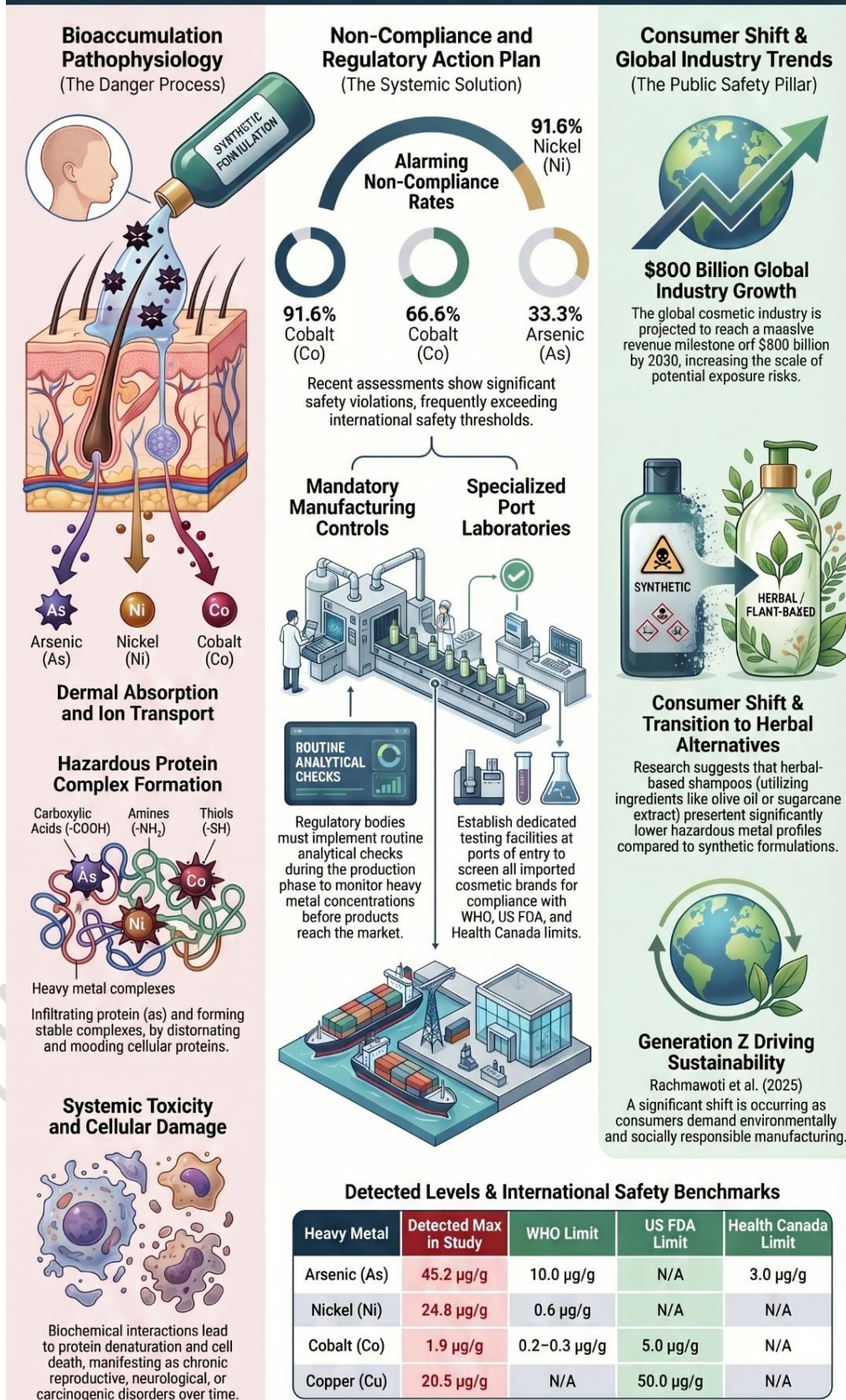
### 3.4.3. Consumer Shift & Awareness: The Public Safety Pillar

The final component of the framework emphasizes the necessity of **public awareness campaigns** to educate consumers on the risks of bioaccumulation from unregulated, inexpensive synthetic shampoos [Gubitosa and Rizzi, 2019; Saleh, 2021]. The proposed transition toward herbal alternatives (Figure 3) is further supported by recent global trends in consumer behavior. Modern consumers, particularly younger generations, are increasingly prioritizing sustainability and social responsibility, driving a shift away from hazardous synthetic chemicals toward safer, natural alternatives [Rachmawati et al., 2025]. Research indicates that herbal-based shampoo formulations carry significantly lower hazardous metal profiles compared to their

synthetic counterparts [Islam et al., 2015; Onna et al., 2021]. Consequently, the framework advocates for a strategic transition toward herbal alternatives as a validated and demonstrably safer option for long-term hair care and public health in the region [Onna et al., 2021; Sukender et al., 2012].

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**Figure 3: Strategic Framework for Public Health Safety and Mitigation of Heavy Metal Risks in Shampoos**



**Figure 3: Strategic Framework for Public Health Safety and Mitigation of Heavy Metal Risks in Shampoos.** This multi-dimensional roadmap integrates experimental findings with systemic solutions: **(1) Bioaccumulation Pathophysiology of toxic ions:** Illustrating the dermal absorption of toxic ions (As, Ni, Co) and their interaction with cellular protein functional groups (-COOH, -NH<sub>2</sub>, -SH), leading to protein denaturation and systemic toxicity. **(2) Regulatory Action Plan:** Proposing mandatory manufacturing controls and the establishment of specialized port laboratories in Aden to address the identified non-compliance rates (91.6% for Ni, 66.6% for Co, and 33.3% for As). **(3) Consumer Shift & Awareness:** Advocating for a transition toward herbal alternatives, supported by 2025 global sustainability trends and the projected USD 800 billion industry growth by 2030.

#### 4. CONCLUSION and RECOMMENDATION

##### 4.1. CONCLUSION

This comprehensive analytical study reveals that a significant majority of the shampoo brands sold in Aden, Yemen, contain **hazardous levels of Arsenic, Nickel, and Cobalt that exceed international safety thresholds.** **The high concentrations of these toxic metals,** particularly in **66.6%** of samples for **Cobalt, 33% for Arsenic** and **91.6% for Nickel,** present a grave **public health concern.** Continuous and cumulative use of these products facilitates the **bioaccumulation** of heavy metals within the **human body** by forming stable complexes with **cellular proteins,** potentially leading to **chronic systemic toxicity, dermatological diseases, and long-term carcinogenic effects.** Research indicates that herbal-based shampoos present significantly lower hazardous metal profiles compared to current synthetic formulations, offering a demonstrably safer option for public consumption.

##### 4.2. RECOMMENDATIONS

Based on the findings of this study, the following strategic actions are recommended to ensure consumer safety:

1. **Manufacturing Oversight:** Regular and rigorous monitoring of heavy metal concentrations must be implemented during the shampoo manufacturing process to prevent contamination.
2. **Regulatory Compliance:** National health authorities should enforce strict adherence to international regulatory guidelines (WHO, US FDA, and Health Canada) for all cosmetic products.

3. **Enhanced Laboratory Screening:** Specialized analytical laboratories should be established at ports of entry to conduct routine screening of imported shampoos before they reach the local market.
4. **Public Awareness:** Educational campaigns are necessary to inform consumers about the toxicological risks associated with cheap, unregulated personal care products.
5. **Shift to Herbal Alternatives:** Consumers and manufacturers should be encouraged to transition toward herbal shampoo formulations, which have been shown to contain significantly lower levels of hazardous heavy metals compared to synthetic brands.

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