Research Journal of Forensic Sciences ISSN 2321–1792
Vol. **14(2),** 22-26, July **(2026)** *Res. J. Forensic Sci.*

Comparative study of Heavy Metals contamination in different Commercial Shilajit Variants: An analytical approach

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Available online at: www.isca.in, www.isca.me
Received 10th April 2026, revised 19th May 2026, accepted 8th June 2026

Abstract

Shilajit is a widely used phyto-mineral substance in traditional medicine; however, concerns regarding heavy metal contamination have raised questions about its safety. The present study aimed to evaluate the presence of cadmium (Cd) and nickel (Ni) in commercially available Shilajit samples using qualitative and quantitative analytical techniques. Four different Shilajit samples were subjected to dry ashing followed by extraction using 4N hydrochloric acid. Qualitative analysis was performed using standard chemical tests, while quantitative determination was carried out using Inductively Coupled Plasma–Mass Spectrometry (ICP-MS). The results indicated that cadmium was below the detectable limit (<0.1 ppm) in all analysed samples. In contrast, nickel was detected in all samples, with concentrations ranging from 0.63 ppm to 1.07 ppm. Although the detected levels were generally within permissible limits, slight variation among samples was observed. The presence of nickel across all samples and variability in its concentration suggest possible influence of environmental factors, geographical origin, and processing techniques. While the immediate risk may be low, the potential for bioaccumulation and long-term exposure cannot be overlooked. The findings highlight the need for strict quality control, standardisation, and regular monitoring of Shilajit products to ensure consumer safety.

Keywords: Shilajit; Heavy metals; Cadmium; Nickel; ICP-MS; Herbal medicine; Forensic Toxicology.

Introduction

Shilajit is a phyto-mineral substance containing both bioactive compounds and trace elements. It is widely used as a supplement in traditional and modern medicine due to its therapeutic effects. It is primarily found in high-altitude mountains, formed after the decomposition of plants and microbial activity. Chemically, Shilajit is a complex material that is a mixture of humic substances, fulvic acid, dibenzo-α-pyrones, amino acids, lipids, and minerals. The composition of shilajit is mainly dependent on geographical origin, environmental conditions, and processing methods, leading to differences in quality and safety[1].

Since shilajit is chemically rich, it is studied extensively for its pharmacological properties. It has shown neuroprotective effects by reducing cell toxicity induced by the amyloid-β in Alzheimer's disease, and has shown strong anti-inflammatory activity, reducing the production of pro-inflammatory markers. This indicates its composition of bioactive compounds, including fulvic acid and other phytochemicals. These properties of shilajit contribute to its use in traditional medicine[2].

However, apart from its benefits, shilajit has been found to contain heavy metals such as Lead (Pb), Nickel (Ni), Cadmium (Cd), Arsenic (As), Mercury (Hg), and copper (Cu). Studies highlighted that many shilajit-containing herbal products are contaminated with several heavy metals exceeding their recommended safety limits due to industrial activities, improper processing techniques, soil pollution, and environmental exposure. These contaminants lead to serious risks like carcinogenicity, neurotoxicity, nephrotoxicity, and hepatotoxicity. Studies suggest that even low-level exposure can have serious effects due to bioaccumulation[3 4].

Several analytical studies have documented the presence of heavy metals in Shilajit and related herbal products, with substantial variations observed among different commercial brands[5,6]. However, inconsistencies in analytical methodologies, sample preparation procedures, and limited comparative evaluations limit the generalisation of these findings. Moreover, many widely consumed Shilajit products remain insufficiently assessed using highly sensitive instrumental techniques capable of trace-level detection.

This study was conducted to compare heavy metal contamination across selected commercially available Shilajit variants. Cadmium and Nickel were selected as target analytes due to their toxicological relevance and frequent detection in herbal formulations. The study employed dry ashing for sample preparation, followed by qualitative chemical screening and quantitative determination using Inductively Coupled Plasma–Mass Spectrometry (ICP-MS). The generated data were evaluated against the World Health Organisation guideline limits to assess the toxicological safety and regulatory compliance of the analysed products.

Materials and Methods

Materials: Shilajit samples from four different herbal brands (SH1RM01, SH2RM02, SH3RM03, and SH4RM04), along with apparatus such as a muffle furnace, crucible, desiccator, and Bunsen burner, as well as glassware including test tubes, beakers, and pipettes, were used for extraction and qualitative analysis. Inductively Coupled Plasma–Mass Spectrometry (ICP-MS) was used for quantitative analysis.

Reagents like 4N hydrochloric Acid (HCL), ammonium hydroxide (NH4OH), sodium sulfide, dimethylglyoxime (DMG), and demineralised water were used for extractions.

Methods: Study Design: The present study was conducted as a comparative analytical study to assess heavy metal contamination in selected commercially available Shilajit variants. The study focused on the detection and quantification of Cadmium (Cd) and Nickel (Ni) using qualitative chemical tests and instrumental analysis using Inductively Coupled Plasma–Mass Spectrometry (ICP-MS).

Sample Selection and Collection: Four commercially available Shilajit resin products (SH1RM01, SH2RM02, SH3RM03, and SH4RM04) were selected for analysis based on their widespread consumer use and market availability. Random sampling was employed to obtain samples from authorised retail sources. Only resin-based Shilajit formulations were included in the study to ensure uniformity in the sample matrix. Newly launched or locally manufactured brands with limited consumer circulation were excluded from the analysis.

Sample Preparation and Extraction of Heavy Metals: Heavy metal extraction from Shilajit samples was carried out using the dry ashing method, which has been widely employed for effective decomposition of organic matrices before trace metal analysis[7]. Approximately 5g of each Shilajit resin sample was accurately weighed using a weighing machine and placed in clean silica crucibles. The samples were initially dried using a hot air oven at 60°C for 25-40 minutes to remove residual moisture and subsequently incinerated in a muffle furnace at 550°C for 1 hour to ensure complete decomposition of organic matter. After cooling to room temperature, the resulting ash was treated with 10 mL of 4 N hydrochloric acid (HCl) to solubilise the metal ions present in the residue. The mixture was gently heated using a Bunsen burner, cooled, and filtered to obtain a clear acid extract suitable for further qualitative and quantitative analysis[8].

Qualitative Analysis of Cadmium (Cd) and Nickel (Ni): Preliminary qualitative analysis was performed to assess the presence of Cadmium (Cd) and Nickel (Ni) in the extracted samples. Cadmium was tested using the sodium sulfide method; however, no characteristic yellow precipitate was observed, indicating the absence or concentration below the detectable limit of Cd^{2+} ions. Similarly, Nickel was analysed using the dimethylglyoxime (DMG) test, but no rose-red precipitate was formed, suggesting that Ni^{2+}ions were not detected under the experimental condition[9]. Blank and standard solutions were processed simultaneously to validate the reliability of the results.

Figure-1: Weighing of a 5g shilaith sample.

Figure-2: Drying of the sample at 60 ° C in a hot air oven.

Figure-3: Dry ashing of the sample.

Figure-4: Crushing and homogenization into a fine powder.

Figure-5: Acid digestion.

Figure-6: Filtration of extract for ICP-MS analysis.

Quantitative Analysis Using Inductively Coupled Plasma–Mass Spectrometry (ICP-MS): Quantitative estimation of Cadmium (Cd) and Nickel (Ni) was performed using Inductively Coupled Plasma–Mass Spectrometry (ICP-MS) operated in kinetic energy discrimination (KED) mode. The acid extracts obtained after dry ashing were used for instrumental analysis, which was selected owing to its high sensitivity, accuracy, and capability for trace-level multi-element detection. The instrument directly provided quantitative concentrations of Cadmium (Cd) and Nickel (Ni) in parts per million (ppm). Calibration standards and quality control samples were analysed alongside test samples to ensure analytical reliability.

Data Interpretation and Safety Assessment: The concentrations of Cadmium (Cd) and Nickel (Ni) obtained from Inductively Coupled Plasma–Mass Spectrometry (ICP-MS) analysis were compared across all four Shilajit samples to evaluate inter-brand variation. The measured values were further assessed in accordance with safety guidelines provided by the World Health Organisation (WHO) and compared with permissible limits established by international regulatory standards to evaluate the toxicological safety of the analysed products[10].

Results and Discussion

The results of the qualitative and quantitative analysis of Cadmium (Cd) and Nickel (Ni) in the analysed Shilajit samples are presented below. All samples were coded to maintain neutrality and avoid brand-specific bias.

Qualitative Analysis: Preliminary qualitative tests for Nickel (Ni) and Cadmium (Cd) were performed using dimethylglyoxime (DMG) and sodium sulfide tests, respectively. No characteristic precipitate was observed in either test, indicating its presence below the detectable threshold of the qualitative method.

Quantitative Analysis: Quantitative determination of Cadmium (Cd) and Nickel (Ni) was carried out using Inductively Coupled Plasma–Mass Spectrometry (ICP-MS). Cadmium (Cd) concentrations in all four samples were found to be below the detectable limit of the instrument (<0.1 ppm). Nickel was detected in trace quantities across all samples, showing minor variation between the coded Shilajit variants.

Table-1: Quantitative concentration of Cadmium and Nickel in coded Shilajit samples.

Sample Code	Cadmium (Cd) (ppm)	Nickel (Ni) (ppm)
SH1RM01	<0.1	1.00
SH2RM02	<0.1	1.07
SH3RM03	<0.1	0.72
SH3RM04	<0.1	0.63

From the Table-1, nickel (Ni) concentrations ranged from 0.63 ppm to 1.07 ppm across the analysed samples. The lowest concentration of Nickel (Ni) was observed in sample SH4RM04, while the highest concentration was recorded in sample SH2RM02. Cadmium (Cd) remained below the quantifiable limit in all samples. Nickel (Ni) concentrations in all samples were within permissible limits; however, continuous exposure may require further toxicological evaluation.

Discussion: The present study demonstrated that cadmium (Cd) was below the detectable limit in all analysed Shilajit samples, whereas nickel (Ni) was consistently detected in trace concentrations across all variants. These findings are in agreement with previous analytical studies, which have reported the presence of trace heavy metals in Shilajit and other herbal formulations due to natural and environmental factors.

A study on thallium contamination in Shilajit reported the occurrence of toxic metals in commercially available samples, highlighting that Shilajit may act as a source of trace elemental exposure[6]. Similarly, spectrochemical investigations using techniques such as ICP-MS, ICP-OES, and LIBS have confirmed the presence of both essential and toxic elements in Shilajit, supporting the detection of nickel observed in the present study[11].

The absence of detectable cadmium in the analysed samples may indicate low environmental contamination or effective processing techniques. However, it should be noted that cadmium may still be present at trace levels below the detection limit of the analytical method. Previous chemical analyses of Himalayan Shilajit have also reported variability in metal concentrations, suggesting that elemental composition is strongly influenced by geographical origin, environmental exposure, and processing conditions[12].

The variation in nickel concentration observed among the samples in the present study further supports this variability. Differences in raw material sourcing, purification methods, and manufacturing practices may significantly affect the final metal content in Shilajit products. These findings are consistent with earlier reports highlighting the lack of standardisation in herbal formulations, which can lead to inconsistent safety profiles[4].

Although the detected concentrations of nickel were within or close to permissible limits, the potential health risks associated with long-term exposure cannot be overlooked. Studies evaluating heavy metals in Ayurvedic products have emphasised that even low concentrations may pose risks when assessed in terms of average daily dose (ADD), particularly with chronic consumption[13]. Furthermore, bioaccessibility studies of toxic metals such as Pb, Cd, Hg, and As have demonstrated that the fraction of metals available for absorption in the human body plays a crucial role in determining toxicity[1].

Therefore, even when metal concentrations are within acceptable limits, factors such as bioavailability, accumulation, and duration of exposure must be considered when evaluating safety. The findings of the present study highlight the need for strict quality control, standardisation, and regular monitoring of Shilajit products to minimise potential health risks.

Conclusion

The present study evaluated the presence of cadmium (Cd) and nickel (Ni) in commercially available Shilajit samples using qualitative and quantitative analytical methods. The results indicated that cadmium was below the detectable limit in all samples, whereas nickel was detected in trace concentrations across all variants. Although the observed levels of nickel were within permissible limits, the potential risk associated with long-term exposure and bioaccumulation cannot be overlooked.

The findings highlight the variability in metal content among different samples, which may be influenced by environmental factors, geographical origin, and processing techniques. Therefore, regular monitoring, standardisation, and strict quality control of Shilajit products are essential to ensure consumer safety.

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