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Pharmaceutico-Analytical Evaluation of a Herbo-Mineral Formulation Containing *Loha Bhasma, Kutaki* and *Haritaki Churna*

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ABSTRACT

Background: *Loha Bhasma* is an important herbo-mineral preparation described in Rasa Shastra and is indicated in *Pandu Roga*. Its pharmaceutical quality depends on proper execution of classical *Shodhana, Bhavana* and *Marana* procedures. The present study was undertaken to prepare *Loha Bhasma* according to classical references and to formulate *Loha Bhasma* along with *Kutaki* and *Haritaki Churna* (LKH).

Aim: To prepare and standardize *Loha Bhasma* according to *Rasa Ratna Samucchaya* and *Rasendra Sara Sangraha* and to prepare the LKH formulation as described in *Rasa Tarangini*.

Materials and Methods: Raw *Loha*, *Hingula, Kutaki, Haritaki* and other required materials were authenticated before processing. *Samanya Shodhana* was performed by seven consecutive *Nirvapa* cycles in *Tila Taila, Takra, Gomutra, Kanji* and *Kulatha Kwatha*, followed by *Vishesha Shodhana* in *Triphala Kwatha. Hingula* was purified by seven *Bhavana* with Nimbu Swarasa. Shuddha Loha was subjected to Marana with purified Hingula and *Kumari Swarasa* through seven *Puta*. *Kutaki* and *Haritaki* were separately powdered and mixed with *Loha Bhasma* to prepare LKH. The prepared *Loha Bhasma* was evaluated by classical *Bhasma Pariksha* and instrumental analytical methods.

Results: Progressive changes in colour, brittleness, texture and weight were observed during pharmaceutical processing. The prepared *Loha Bhasma* fulfilled *Rekhapurnatva, Varitaratva, Unnama and Nischandratva*. The final yield was 463.3 g from 450 g of *Shuddha Loha* (102.96%). Powdering of *Kutaki* and *Haritaki* gave a combined yield of 85.71%, and the final LKH formulation was obtained as a homogeneous light-brown powder. Analytical evaluation showed a submicron particle population, characteristic Fe–O bands in FTIR, Hematite (Fe2O3) as the predominant crystalline phase by XRD, and an iron content of 67.48% by XRF.

Conclusion: The classical pharmaceutical procedures successfully transformed *Loha* into a fine, non-lustrous *Bhasma* fulfilling the desired classical characteristics. The LKH formulation was prepared as a homogeneous blend and the analytical

findings supported its pharmaceutical characterization.

Keywords: *Loha Bhasma, Shodhana, Marana, Kutaki, Haritaki*, LKH, Ayurvedic Pharmaceutics.

INTRODUCTION

Loha Bhasma is one of the extensively used mineral preparations in *Ayurveda* and has been described for the management of *Pandu Roga* and disorders associated with *Rakta Dhatu*. Its pharmaceutical preparation involves systematic purification and incineration to transform raw metallic iron into a therapeutically acceptable preparation[1].

According to *Rasa Shastra*, raw *Loha* is not administered directly because of its hardness, poor digestibility and limited biological availability. *Samanya Shodhana* and *Vishesha Shodhana* are therefore performed to reduce metallic hardness, increase brittleness and facilitate subsequent processing. Repeated *Bhavana* and controlled *Puta* during *Marana* further transform purified *Loha* into a fine, soft and non-metallic *Bhasma*.

Rasa Tarangini describes the use of *Loha Bhasma* in combination with *Kutaki* and *Haritaki* for *Pandu Roga*.[2] Since detailed pharmaceutical documentation of this combination is limited in the present source material, the current study was undertaken to prepare *Loha Bhasma* by classical procedures and formulate LKH, documenting pharmaceutical observations, yields and analytical characteristics.

AIM AND OBJECTIVES

1. To prepare *Loha Bhasma* by adopting classical *Shodhana* and *Marana* procedures.
2. To document the pharmaceutical observations and process-related changes during each stage of preparation.
3. To evaluate the prepared *Loha Bhasma* by classical *Bhasma Pariksha*.
4. To prepare the LKH formulation using standardized *Loha Bhasma*, *Kutaki* Churna, and *Haritaki* Churna.
5. To record the pharmaceutical yield and organoleptic characteristics of the prepared formulation.

MATERIALS & METHODS

Procurement of raw material

Three samples of raw *Loha* were procured from different sources. Raw *Hingula*, *Kutaki*, *Haritaki* and *Triphala* were procured from Shree Ram Herbals, Jaipur. Sesame oil (*Tila Taila*), *Takra*, *Gomutra*, and *Kulatha* were obtained from the local market of Jodhpur. Fresh *Kumari* leaves were collected from the herbal garden of Dr. S.R. Rajasthan Ayurved University, Jodhpur.

Authentication of raw drug

All the raw materials were authenticated by experts of the Departments of *Rasa Shastra and Bhaishajya Kalpana* and *Dravyaguna* before pharmaceutical processing.

Composition of LKH

The present study was a pharmaceutical study conducted in the Department of *Rasa Shastra and Bhaishajya Kalpana* with the objective of preparing and standardizing *Loha Bhasma* and formulating *Loha Bhasma* along with *Kutaki* and *Haritaki* Churna (LKH) according to the classical Ayurvedic references.

Table 1. The formulation of the composition is given in Table.

S. No.	Ingredient	Quantity
1.	*Loha Bhasma*	135 mg
2.	*Kutki Churna*	2932.5 mg
3.	*Haritaki Churna*	2932.5 mg

The pharmaceutical preparation of the formulation was carried out according to classical Ayurvedic methods and included the following sequential stages:

1. *Samanya Shodhana* of *Loha*
2. *Vishesha Shodhana* of *Loha*
3. *Marana* of *Sudha Loha*

4. Preparation of *Kutki* & *Haritaki* Churna
5. Preparation of the final formulation containing *Loha Bhasma*, *Kutki* Churna, and *Haritaki* Churna (*LKH*)

1. *Samanya Shodhana* of *Loha*

- **Preparation of *Kanji*[3]**

Kanji was prepared according to the method described in *Rasa Ratna Samuccaya*. Rice grains were cooked with fourteen parts of water to obtain *Manda*, which was transferred into a fumigated porcelain jar containing a small quantity of cooked rice. The container was sealed using *Kapad-Mitti* and allowed to ferment for 40 days. The prepared *Kanji* was transparent in appearance with a strong pungent odour and an acidic pH of 3, indicating successful fermentation.

- **Preparation of *Kulatha Kwatha*[4]**

Kulatha Kwatha was prepared according to *Sharngadhara Samhita*. Cleaned *Kulattha* seeds were soaked overnight and boiled with sixteen parts of water until the volume was reduced to one-eighth. The decoction was filtered through a clean cotton cloth before use. The prepared *Kwatha* was dark brown in colour with a characteristic odour and a pH of 6.58.

- ***Nirvapa* process of *Loha*[5]**

Samanya Shodhana of *Loha* was performed by *Nirvapa Vidhi* as described in *Rasa Ratna Samuccaya*. Raw *Loha* (500 g) was heated to red-hot condition and successively quenched seven times each in *Tila Taila*, *Takra*, *Gomutra*, *Kanji*, and *Kulatha Kwatha*, using fresh media for every quenching cycle. During each cycle, temperature, time required for heating, and changes in weight were recorded. Appropriate safety precautions were followed throughout the procedure.

- **Pharmaceutical Observations**

Repeated heating and quenching produced marked changes in the physical characteristics of *Loha*. The metallic lustre gradually disappeared, and the material became brittle, porous, and progressively converted into fine powder. The colour of *Loha* changed from metallic grey to deep brown after successive quenching cycles. Each quenching medium also exhibited characteristic changes in colour, odour, and consistency, confirming interaction between the heated metal and the processing media. The final weight of *Loha* after *Samanya Shodhana* was (483.8 g).

2. *Vishesha Shodhana* of *Loha*

- **Preparation of *Triphala Kwatha*[6]**

Triphala Kwatha was prepared according to the method described in *Sharangadhara Samhita*. Equal parts of *Haritaki*, *Amalaki*, and *Bibhitaki* were coarsely powdered (*Yavakuta*), soaked overnight in water, and boiled until the volume was reduced to one-fourth. The decoction was filtered through a clean cotton cloth and freshly prepared for each cycle of *Vishesha Shodhana*. The prepared *Kwatha* was dark brown in colour with a characteristic odour, *Kashaya-Amla* taste, and a pH of 3.47.

- ***Nirvapa* process of *Loha*[7]**

Samanya Shodhita Loha (483.8 g) was subjected to *Vishesha Shodhana* by repeated ***Nirvapa*** in freshly prepared *Triphala Kwatha*, following the procedure described in *Rasa Ratna Samuccaya*. The *Loha* was heated to red-hot condition and quenched seven consecutive times in *Triphala Kwatha*. Changes in weight and pharmaceutical characteristics were recorded after completion of the procedure.

Pharmaceutical Observations

During repeated heating, a reddish flame and crackling sound were observed. Fine particles of *Loha* adhered to the ladle and a small quantity was dispersed during quenching. The colour of *Loha* gradually changed to dark black, while the *Triphala Kwatha* changed from brown to greyish-black. Boiling and occasional overflow of the decoction were observed during quenching. The final weight of *Loha* after *Vishesha Shodhana* was **455.4 g**, with an overall yield of **91.08%**.

3. *Marana* of *Sudha Loha*

- ***Shodhana* of *Hingula*[8]**

Hingula Shodhana was carried out according to the procedure described in

Rasa Tarangini. *Ashuddha Hingula* (402.61 g) was finely powdered in a *Khalva Yantra* and subjected to seven successive *Bhavana* using **200 mL of freshly prepared *Nimbu Swarasa***, which was obtained from **500.16 g of fresh lemons**. The purification process was completed over **9 days**, and levigation was continued in each cycle until complete absorption of the *Swarasa*. After completion of seven *Bhavana*, the processed material was washed thoroughly with warm water (*Prakshalana*), dried under sunlight, and stored in an airtight glass container for further pharmaceutical processing.

Results

Progressive pharmaceutical changes were observed during *Shodhana*. The brown crystalline *Hingula* gradually transformed into a fine reddish powder. The material remained insoluble in *Nimbu Swarasa*, and no effervescence or heat evolution was observed during levigation. The processed material became progressively adhesive during successive *Bhavana*, while the quantity of *Nimbu Swarasa* required for each subsequent *Bhavana* gradually decreased. After drying, the purified *Hingula* acquired a uniform reddish-orange colour with complete loss of its crystalline lustre, indicating successful purification. The final weight of purified *Hingula* was **407.61 g**, showing a net weight gain of **5.0 g** with a percentage yield of **101.24%**.

- **Preparation of *Kumari Swarasa*[9]**

Fresh *Kumari* (*Aloe vera*) leaves were collected from the herbal garden and processed according to the method described in *Sharangadhara Samhita*. After washing and removing the thorny margins, the pulp was separated, triturated, and filtered to obtain fresh *Kumari Swarasa*. A total of **1980 mL** of *Swarasa* was obtained from **3440.92 g** of fresh *Kumari* leaves (57.54% yield). The prepared *Swarasa* was watery in appearance with a bitter taste and characteristic pungent odour.

***Puta* preparation of *Shuddha Loha*[10]**

Marana of *Shuddha Loha* was carried out according to the procedure described in *Rasendra Sara Sangraha*. Purified *Loha* (450 g) was mixed with **1/12th part of purified *Hingula*** and triturated with sufficient quantity of freshly prepared *Kumari Swarasa* to obtain a homogeneous paste. The paste was converted into uniform *Chakrikas*, dried completely, sealed in *Sharava Samputa* using *Kapad-Mitti*, and subjected to **seven consecutive Puta** in a horizontal electric muffle furnace. During each *Puta*, the temperature was maintained at **750°C for 1 hour**[11], followed by self-cooling (*Swangasheeta*). After each cycle, the material was triturated again with *Hingula* and *Kumari Swarasa*, and the same procedure was repeated until completion of seven *Puta*.

Results

Progressive pharmaceutical changes were observed after each *Puta*. The colour of the *Chakrikas* gradually changed from greyish-black to deep purple (*Pakwa Jambuphala Varna*), while the texture became progressively softer and smoother. *Rekhapurnata* was observed from the third *Puta* onwards, whereas *Varitaratva* increased gradually with successive incineration cycles and became complete after the seventh *Puta*. The metallic lustre (*Chandrika*) progressively disappeared, and magnetic attraction was markedly reduced after repeated *Puta*. After completion of the seventh *Puta*, the prepared *Loha Bhasma* fulfilled all the classical *Bhasma Pariksha*, including *Rekhapurnata*, *Varitaratva*, and *Unnama Pariksha*. The final yield of *Loha Bhasma* was **463.3 g** from **450 g** of *Shuddha Loha*, corresponding to a percentage yield of **102.96%**.

4. Preparation of *Kutki* & *Haritaki* Churna[12]

Equipment

Pulverizer, 85-mesh sieve, weighing machine, steel tray.

Method

Kutki and *Haritaki* were manually cleaned to remove visible impurities, washed thoroughly with tap water, and dried completely under sunlight. The dried drugs were pulverized in Nagarjun Rasayanshala using a pulverizer. Initially, coarse powder was obtained, which was further pulverized to produce a fine powder. The powder was passed through an 85-mesh sieve to obtain a uniform particle size. The sieved powder was collected and stored in clean, airtight glass containers until further use.

Precautions

- The drugs were ensured to be completely free from moisture before powdering.
- A face mask was used during grinding to prevent irritation caused by fine powder.

Results

Initial weight of Kutki and Haritaki was 3.5 kg each. After pulverization and sieving, 6 kg of fine powder was obtained with a percentage yield of 85.71%.

5.Preparation of *LKH*[13]

The final *LKH* formulation was prepared by thoroughly mixing *Loha Bhasma* (135 mg), *Kutaki Churna* (2932.5 mg) and *Haritaki Churna* (2932.5 mg) to obtain a homogeneous blend. The prepared formulation was stored in airtight glass containers for further evaluation.

Analytical Evaluation

The analytical studies were carried out Drug Testing Laboratory, Dr. Sarvepalli Radhakrishnan Rajasthan Ayurved University, Jodhpur and Jeevanrekha Analytical Services (OPC) Private Limited, Deolai, Aurangabad.

Loha Bhasma was evaluated for organoleptic characteristics, classical Bhasma Pariksha, physicochemical parameters, particle size, FTIR, XRD and XRF. HPTLC fingerprinting of the herbal components of LKH was also recorded.

Results

Table 2.-Pharmaceutical Yield

Pharmaceutical procedure	Initial weight	Final weight	Yield (%)
Samanya Shodhana of *Loha*	500 g	483.8 g	96.76
Vishesha Shodhana of *Loha*	483.8 g	455.4 g	91.08
Shodhana of *Hingula*	402.61 g	407.61 g	101.24
Marana of *Loha*	450 g	463.3 g	102.96
Powdering of *Kutaki* and *Haritaki*	7 kg	6 kg	85.71

A gradual reduction in the weight of *Loha* was observed after *Samanya* and *Vishesha Shodhana*, whereas a slight increase in weight was noted after *Marana*. The increase may be attributed to repeated *Bhavana* with *Kumari Swarasa* and incorporation of purified *Hingula* during successive *Puta*.

Table 3.-Organoleptic and Classical Bhasma Evaluation

Parameter	Loha Bhasma	LKH
Appearance	Fine powder	Fine homogeneous powder
Colour	Purple (Pakwa Jambuphala Varna)	Light brown
Touch	Soft and smooth	—
Odour	Odourless	Characteristic
Taste	Tasteless	Bitter and astringent (Tikta, Kashaya)

Loha Bhasma was positive for Rekhapurnatva, Varitaratva, Nischandratva and Unnama Pariksha.

Table 4: Physico-chemical Evaluation of *LKH* Formulation

S. no.	Plant name	Mobile phase	Rf value at 254nm	Rf value At366nm	Biomarker
1.	*Pichrohiza Kurroa*	Methanol, formic acid, ethyl acetate 0.8:1.2:12	0.52	0.89,0.34	Picroside
2.	*Terminalia Chebula*	Methanol, formic acid, ethyl acetate, toluene0.3:1.2:4.3:4.3	0.64,0.77	0.63,0.78	Tannic acid

Table 5: HPTLC Fingerprint Profile of LKH

S. No.	Test Parameter	Result	Unit
1	pH value	5.5	-
2	Loss on Drying	7.14	%
3	Total Ash	11.02	%
4	Acid insoluble Ash	2.45	%
5	Water soluble extractive	53.8	%
6	Alcohol soluble extractive	43.09	%

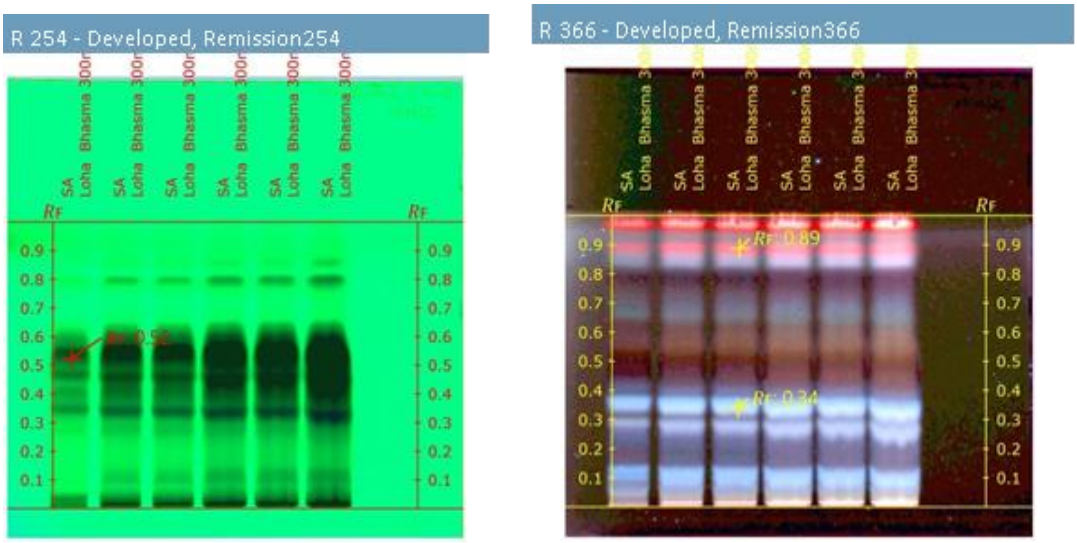

Figure 1,2.-HPTLC plate showing kutki (Picroside) Rf at 254nm,366nm

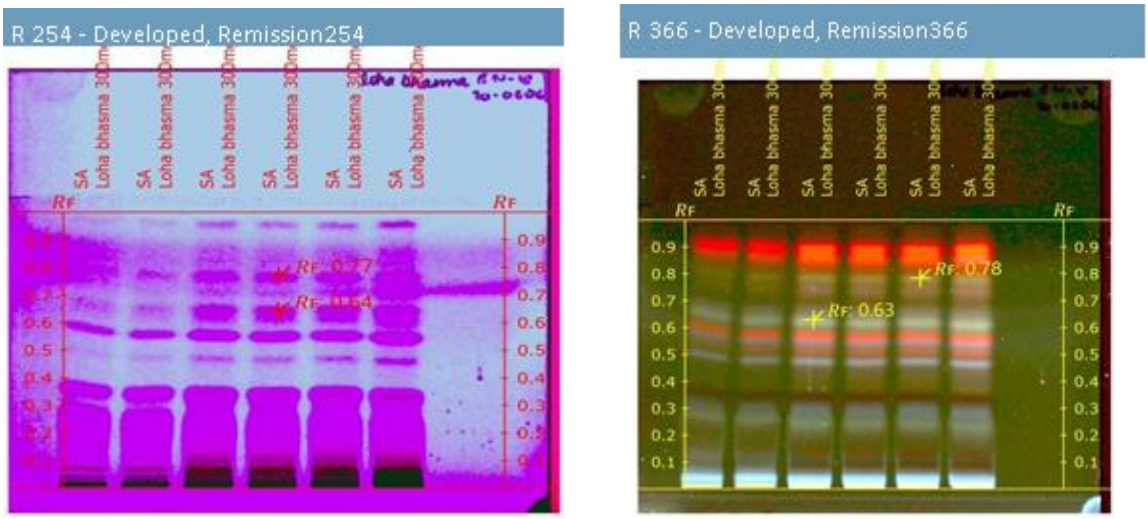

Figure 3,4-HPTLC plate showing *Haritaki* (Tannic acid) Rf at 254nm,366nm

Particle Size, FTIR, XRD and XRF

Particle size analysis using BIOVIS PSA 2000 revealed a total of 18,834 particles, including 18,341 single particles and 493 agglomerates. The d_{10}, d_{50} and d_{90} values of single particles were 0.15, 0.27 and 0.44

µm, respectively, while those of agglomerates were 0.18, 0.47 and 0.92 µm, indicating a predominantly submicron and finely dispersed particle population.

FTIR analysis showed characteristic absorption bands at 3571–3753 cm^{-1} corresponding to O–H stretching vibrations, 2374 cm^{-1} related to atmospheric CO_2, and bands at 552 and 474 cm^{-1} corresponding to Fe–O stretching and bending vibrations, respectively. The Fe–O bands support the formation of iron oxide during the pharmaceutical processing of *Loha Bhasma*.

XRD analysis demonstrated a well-defined crystalline phase, with major diffraction peaks at 2θ values of 28.087°, 38.820° and 41.641°, which were identified as Hematite (Fe_2O_3) having a trigonal crystal structure. The maximum peak intensity was 86.8%, indicating good crystallinity and confirming Hematite as the predominant crystalline phase.

XRF analysis further demonstrated an iron-rich elemental composition, with Fe constituting 67.48% of the sample. Other detected elements included Al (1.19%), Si (5600 ppm), P (2370 ppm), S (580 ppm), Cl (980 ppm), Ca (6280 ppm), Mn (2560 ppm), Ti (500 ppm), Cr (102 ppm), Cu (162 ppm), Zn (94 ppm) and Sr (15 ppm). Overall, the analytical findings indicate that the prepared *Loha Bhasma* possessed a fine submicron particle size, characteristic Fe–O functional groups, crystalline Hematite as the major phase, and a predominantly iron-based elemental composition, supporting its pharmaceutical characterization and standardization.

RESULTS

The prepared *Loha Bhasma* showed progressive changes in colour, texture, brittleness and weight during *Shodhana* and *Marana*. The final *Loha Bhasma* yield was 463.3 g from 450 g of *Shuddha Loha* (102.96%) and fulfilled *Rekhapurnatva, Varitaratva, Nischandratva* and *Unnama Pariksha*. The LKH formulation was obtained as a fine, homogeneous light-brown powder. Physicochemical analysis showed pH 5.5, loss on drying 7.14%, total ash 11.02% and acid-insoluble ash 2.45%. HPTLC fingerprinting demonstrated characteristic marker peaks corresponding to Picroside in *Kutaki* and Tannic acid in *Haritaki* at 254 and 366 nm. Particle size analysis revealed predominantly submicron particles with d_{50} of 0.27 µm for single particles. FTIR showed characteristic Fe–O bands, while XRD confirmed Hematite (Fe_2O_3) as the predominant crystalline phase. XRF analysis demonstrated an iron content of 67.48%, supporting the pharmaceutical and analytical characterization of the prepared formulation.

DISCUSSION

The present pharmaceutical study demonstrated that systematic application of classical Shodhana and Marana procedures produced progressive physical transformation of Loha. Repeated Nirvapa reduced metallic lustre and increased brittleness and friability, thereby facilitating subsequent processing. Vishesha Shodhana in Triphala Kwatha further altered the physical characteristics and supported preparation of Loha for Marana.

During Marana, repeated Bhavana with Kumari Swarasa and incorporation of purified Hingula followed by successive Puta resulted in progressive changes in colour, texture, fineness and magnetic property. Completion of Rekhapurnatva, Varitaratva, Unnama and Nischandratva after the final Puta indicated attainment of the classical Bhasma characteristics.

The analytical findings complemented the classical evaluation. The submicron particle size indicated a fine particulate nature of the prepared Bhasma. FTIR bands in the lower wavenumber region were consistent with Fe–O vibrations, while XRD identified Hematite as the predominant crystalline phase. XRF demonstrated that iron was the major elemental constituent. The physicochemical values and HPTLC fingerprinting further provided quality

characteristics for the prepared material and the herbal components of LKH.

CONCLUSION

The present study successfully prepared Loha Bhasma through classical Shodhana and Marana procedures and subsequently formulated LKH with Kutaki and Haritaki Churna. Progressive pharmaceutical changes and satisfactory yields were observed during processing. The prepared Loha Bhasma fulfilled the evaluated classical Bhasma Pariksha and demonstrated fine submicron particles, characteristic Fe–O vibrations, Hematite as the predominant crystalline phase and an iron-rich elemental composition. The LKH formulation was obtained as a homogeneous light-brown powder, while HPTLC fingerprinting demonstrated characteristic markers of the herbal components. The findings provide pharmaceutical and analytical characteristics of the prepared Loha Bhasma and LKH formulation.

Declaration by Authors

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Conflict of Interest: The authors declare no conflict of interest.

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