Original Research Article

A Comprehensive Analysis and Validation of *Trividha Putapaka Marita* and *Hingula Marita Lauha Bhasma* .

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Abstract:

Introduction: *Rasashastra,* is an ancient discipline focusing on the medicinal properties of metals.. *Lauha* is a metal , which primarily plays a vital role in addressing various disorders according to *Ayurvedic* literature.

Purpose: The primary objective of this research is to validate the production methods of *Lauha Bhasma influenced* by *Rasashastra*. Validation, a critical process ensuring consistent and reliable outcomes, is undertaken to analyse the medicine produced following specified protocols. The study aims to detect variations in *Lauha Bhasma* after a specific number of *Puta* cycles, employing two distinct methods of *Marana.*

Method: The research employs a meticulous approach, adhering to classical *Ayurvedic* texts, for the *Shodhana (*purification) and *Marana* (incineration) processes of *Lauha.* Two methods, *Trividha Putapaka Marita* and *Hingula Marita,* are followed. The study also involves detailed physiochemical analyses, including *Bhasma Pariksha* and Energy Dispersive X-ray Spectroscopy (EDS), to assess the quality and composition of *Lauha Bhasma.*

Result: The outcomes of the study reveal distinct characteristics in *Lauha Bhasma* A and B, derived from different *Marana* methods. Organoleptic, *Bhasma Pariksha,* and physiochemical analyses showcase variations in colour, texture, and elemental composition. EDS analysis emphasizes differences in iron content, providing valuable insights into the impact of *Marana* processes on the final product.

Conclusion: These analyses reveal variations in iron content, ash content, water solubility, pH, and moisture content between the two samples, *Lauha Bhasma* A and *Lauha Bhasma* B. The findings underscore the significance of precise preparation methods in Ayurvedic medicine, offering insights for practitioners and researchers alike.

Keywords :

Introduction-

Rasashastra is a subject that deals with metals and their medicinal properties. It describes the use of metals in a purified form called *Bhasma.* Their use reduced the quantity of dose administered while increasing the palatability, tissue availability, and shelf-life of the medications.[(1)] The descriptions of mercury processing procedures dominate *Rasa Shastra.* As a result, the word "*Rasa*" is regarded a synonym for "mercury." Aside from mercury, *Rasa Shastra* describes the value of metals such as gold, silver, copper, tin, lead, and iron, as well as salts, corals, seashells, and feathers. Because mineral and metal elements in their natural form can be detrimental to the human body, they require particular preparation techniques before they can be utilized in therapies. A wide range of *Shodhana* (purification and/or detoxification) methods are prescribed for metals, minerals, and other substances in different classics of *Rasashastra.*[(2)] These methods not only detoxify the raw material by chemical transformations but also enhance their therapeutic potentials.

Lauha (iron) is a vital component of the human body that is required for the treatment of numerous diseases as well as for physiological functioning. *Lauha* is widely used in *Ayurvedic* literature for the treatment of many disorders such as *Pandu* (Anaemia), *Shotha* (Oedema), *Kamala* (Jaundice), and so on.[(3)] *Lauha Bhasma* has been used in *Ayurveda* for a long time, although it was originally intended for therapeutic purposes during the reign of *Nagarjuna.* Collections, purification, incineration, preparation of different varieties of *Lauha Bhasma* are described in various classical texts of *Ayurveda* in detail.

Validation is the crucial process of confirming the reliability of established procedures through scientific grounding, ensuring consistent product delivery. It has gained global acceptance, often replacing standardization. In the realm of pharmaceuticals influenced by *Rasa* Classics, validation marks the culmination, requiring the navigation of numerous milestones. While formulations for classics are already standardized, validation involves affirming existing processes. Once a process consistently produces unvarying results, it attains validity.[(4)] Process validation ensures reproducibility, even in the hands of an unskilled operator,

and aids in meticulous record-keeping for complex and lengthy procedures. The aim of this study is to thoroughly analyze the medicine produced following the provided protocol, utilizing the specified standard raw materials. The objective is to detect any variations that may arise in *Lauha Bhasma* after a specific number of *Puta* cycles during its manufacturing process by two methods of Marana.

Aims and objectives-

Aim-To compare physiochemical analysis of two different *Lauha Bhasma* preparations and to obtain best suitable method of *Lauha Bhasma* preparation.

Objective- To make two different varieties of *Lauha Bhasma*-

1. *Lauha Bhasma A: Trividha Putapaka Marita Lauha Bhasma*
2. *Lauha Bhasma B: Hingula Marita Lauha Bhasma*

Materials and methods- *Shodhana* of *Lauha* was done based on the method described in *Rasaratna Samuchaya*. *Marana* of *Lauha* was done by methods described in *Rasendra Sara Sangraha*(*Trividha Putapaka* method) and *Ayurveda Prakash* (*Hingula Marita* method).

Procurement of raw drugs: The raw *Teekshna Lauha* and *Shodhita Hingula* were sourced from department of *Rasashastra*.Selection of raw material was done according to *Grahya Lakshana* mentioned in texts. All other materials required for the process of *Shodhana* and *Marana* were procured from local market.

Lauha Shodhana-According to *Rasaratnasamuchaya, Samanya* and *Vishesha Shodhana* of *Lauha* was done.

Samanya Shodhana of *Lauha*-[5]

Principle: *Nirvapa* (heating, followed by quenching) Material-Raw *Teekshna Lauha (*thin metal sheet) -2 kg

Media: 1. *Tila Taila* (*Sesamum indicum Linn.* Oil) 2. *Takra* (butter milk) 3. *Gomutra* (cow urine) 4. *Kanji* (sour gruel made from Oryza sativum Linn.) 5. *Kulattha Kwatha* (decoction of seeds of *Dolichos biflorus Linn*.)-Q.S. Duration: 6 days. Equipments: *Patra (Stainless* steel vessels), measuring vessel, weighing scale, gas stove (kitchen type) etc. Duration-23 hours.

Procedure-A thin metal sheet of *Teekshna Lauha* was heated to a high temperature and submerged in *Tila taila. It* was reheated and then again dipped in a new sample of *Tila Taila*. The process is repeated seven times. The same metal sheet was then similarly processed by being heated and quenched in liquids as *Takra* (buttermilk), *Gomutra* (cow urine), *Kanji* (sour gruel), and *Kulattha Kwath*(*Dolichos biflorus*). So, dipping in five different liquids seven times total of 35 times. A new, gravimetrically identical amount of media was taken each time to quench the *Teekshna Lauha*. Each time, the *Shodhita Lauha* weight and melting time were recorded. Observations made during the *Shodhan* of *Lauha have* been meticulously recorded, both in *Lauha* and in the media. The quantity obtained after *Samanya Shodhana* of *Lauha* was 2500 gm.

Observations- Following the *Nirvapa* process, *Lauha* was converted into granular form. Color of *Tila taila turned* blackish brown and brownish color of *Lauha* became black and its metallic luster was lost. weight of *Lauha* was found increased. Color of *Lauha* became black to blackish gray after *shodhana* in *Takra.* During quenching, liquid media emitted dense fumes, accompanied by a vigorous boiling sound. A pungent smell was perceived during quenching in *Gomutra.* In the *Kanji* process of *Shodhana,* the color of *Lauha t*ransitions from brown to a blackish-brown. The *Lauha* became increasingly brittle and underwent a transformation into a coarser powder form. Color of *Kulattha Kwatha* became brown to bluish brown and its consistency became thicker. There was reduction in weight of *Lauha* after *Shodhana* in each media except in *Tila taila.*

Vishesha shodhana of *Lauha*-[6]

Then, according to *Rasaratnasamuchaya's* classical reference, *Vishesha Shodhana* with *Triphala kwatha* was performed.

Principle: *Nirvapa.* Materials-*Samanya Shodhita Lauha* - 2500 gm, Media: *Triphala kwatha*- Q.S. Equipments: Gas burner, cylinder, lighter, iron ladle, iron rod, spatula, measuring cylinder, weighing machine, etc.

Procedure -The *Samanya Shodhita Lauha* was heated on a gas stove. Metal was quenched in *Triphala Kwatha.* The process was repeated for seven times. The quantity obtained after *Vishesha Shodhana* of *Lauha* was 2450 gm.

Observations-After the *Shodhana* process, the previously silver-grey tint of the *Lauha* turned to a deep black color, and the iron sheet underwent a transformation into a coarse powder. Upon completion of the *Shodhana* process, the color of the *Lauha* transitioned from its original silver-grey to a profound black shade. Increase in weight was observed following *Shodhana.* An audible minor explosion was observed when introducing the *Lauha* into the *Triphala* decoction. 2 % of weight loss was observed. This whole procedure makes *Lauha* more brittle and leads to partial conversion into coarse powder form.

Marana* of *Lauha-

Shodhita Lauha was divided in two parts of 1000 gm each. Marana of *Shuddha Lauha* was done by two different

methods.

1. *Maran* of *Lauha* by method mentioned in *Rasendra Sara Sangraha (Trividha Putapaka* method)- ***Lauha Bhasma* A**

The *Marana* process was accomplished in three steps: *Bhanupaka, Sthalipaka* and *Putapaka*. Materials: Ingredients required for preparation of *Lauha Bhasma* were as follows

1 *Shodhita Lauha Churna.* – 1000 gm

2 *Triphala Kwatha.* – Q.S.

Equipment's: Weighing machine, *Khalva yantra,* earthen *Sharav Samputa,* knife, *multani mitti,*

Cotton cloth, utensils, mixer and *Vanyopala, etc.*

A. *Bhanupaka* for *Lauha Marana*-[7,8]

Following the completion of the *Samanya* and *Vishesha Shodhana* steps, the *Shodhita Lauha Churna* was placed in a tray, and *Triphala Kwatha* was added until *Lauha Churna* was entirely immersed in it. Following that, the mixture was uniformly mixed together and dried in the sunlight till it becomes entirely dry. The process was repeated six times more (for a total of seven times). Quantity of *Lauha churna* obtained after *Bhanupaka* was 1050 gm.

Observations- Following *Bhanupaka,* appearance of *Lauha* changed to suspended solid granules.

B. *Sthalipaka* for *Lauha Marana*-

Dried *Lauha Churna* was transferred into an iron container for the *Sthalipaka* procedure, and *Triphala Kwatha* was added to the same iron container. Following that, the iron container was placed on the heating equipment and heated until the liquid content of *Triphala Kwatha* was completely evaporated. This method was repeated six times more (for a total of seven times). Quantity of *Lauha churna* obtained after *Sthalipaka* was 1080 gm.

Observations- During *Sthalipaka* there were reduction in appearance of *Lauha churna*.

C. Putapaka for *Lauha Marana*-

During the *Putapaka* phase, *Lauha Churna* from the *Sthalipaka* procedure was triturated with *Triphala Kwatha*. The triturated material was then used to produce pallets (*chakrika),* which were then dried until the moisture content was totally evaporated. Following that, dried *Chakrika* were collected and evenly distributed in *Sharava* and *Sharava samputa* was done. The sealed *Sharava samputa* were then placed in the *Putayantra,* subjected to heat by *Gajaputa.* After *Swangsheeta Samputa* was removed and opened, followed by taking out *Lauha churna* from it. The *Putapaka* procedure described above was performed up to 30 times to get *Lauha Bhasma* of the required quality. After each *Puta, Bhasma Pariksha* was carried out according to *Ayurvedic* texts.

Observations-After the first five *Puta,* the coloration of the *Lauha Chakrika* was blackish brown, and they were quite hard in touch. A metallic luster was detected on the surface of the pellets. Pellets turned purple after the eleventh *Puta,* but they remained firm. After the sixteen *Puta,* the tint changed to purple, the pellets became soft, and the metallic luster was no longer discernible. After the thirty *Puta,* the color of *Lauha Bhasma* was purple (*Pakwa Jambu Phala Varna*), soft and smooth to the touch, and metallic lusterless. *Bhasma pariksha* was conducted and it was observed that *Bhasma* was passing all classical tests. Quantity of *Lauha Bhasma* A obtained after 30 *Puta* was 358 gm.

2. *Maran* of *Lauha* by method mentioned *in Ayurveda Prakash (Hingula Marita Lauha Bhasma)*-***Lauha Bhasma B*** [9]

Materials: Ingredients required for preparation of *Lauha Bhasma* were as follows

1 *Shodhita Lauha Churna*-1000 gm

2. *Shodhita Hingula* – 80 gm

3. *Kumari Swarasa (Aloe barbadensis miller-* fresh juice*)* – Q.S.

Equipments: Weighing machine, *Khalva yantra*, earthen *Sharav samputa,* knife, *multani mitti,* cotton cloth, utensils, mixer and *Vanyopala* ,etc.

Procedure-First, 1000 gm of *Shodhita Lauha Churna* was ground followed by addition of 80 gm of *Shodhita Hingula* and triturated with *Kumari swarasa* for six hours to make a homogeneous mixture. The *Chakrika* was then formed into 3 cm diameter 0.3 cm thick and dried in the shade for 48 hours. The dried pellets were then placed in *Sharawa samputa* and *Sandhibandhan* was done and, subjected to heat by *Gajaputa.* After *Swangsheeta samputa* was removed and opened, followed by taking out *Lauha churna* from it. The *Puta* procedure described above was repeated to get *Lauha Bhasma* of the required quality. After each *Puta Bhasma Pariksha* was carried out according to *Ayurvedic* texts. After 7 such Puta *Varitara Bhasma and Bhasma siddhi lakshanas* were obtained and final weight was 730 gm.Colour of *Lauha Bhasma* B was dark violaty Brown.

Observations- After each *Puta,* the color of the *Lauha* shifted from a brown to a more pronounced dark brown-violet shade. The pellets exhibited a fragile nature, easily breaking upon slight contact, and most of them presented a black appearance. Following the third round of *Puta,* a noticeable change to a reddish-brown color was witnessed. Remarkably,

the weight of the *Bhasma* consistently decreased after each subsequent *Puta* cycle. The Chakrika underwent a progressive softening in texture with successive *Puta* cycles. Furthermore, it was noted that the quantity of cow-dung cakes used in the process was systematically reduced as the *Puta* cycles advanced.

The samples (*Ashuddha Lauha, Shodhita Lauha* and *Lauha Bhasma* A and B) were subjected to EDS (energy dispersible X ray diffraction) analysis.

Results-

Table 1 indicates organoleptic characters and *Bhasma Pariksha* of different samples of *Lauha Bhasma.*

Table 1- organoleptic characters and B*hasma pariksha* of *Lauha bhasma* A and B

parameter	*Lauha Bhasma A*	*Lauha Bhasma B*
Shabda	Absent	Absent
Sparsha	Rough, no course particles	Soft
Roopa	Blackish brown,	Dark violate Brown.
Rasa	Tasteless	Tasteless
Gandha	Non specific	Not specific
Snigdhatwa	*Alpa snigdha*	*Alpa snigdha*
Nischandratwa	No metallic luster	No metallic luster
Rekhapurnatwa	Present	Present
Varitaratwa	Absent	Present
Unama	Absent	Present

Table 2 shows physiochemical analysis of different *Lauha* and *Lauha bhasma* samples.

Table 2- Physio-chemical Analysis of *Ashodhit,Shodhita and Lauha Bhasma –A and B*

Sr No	Lauha sample	Total Ash content (%)	Acid insoluble extractive (%)	Water soluble ash (%)	pH	LOD @110 C (%)
1	*Ashodhit Lauha*	102.21	BDL	0.06	4.56	0.04
2	*Shodhita Lauha*	47.36	33.52	6.11	5.86	1.32
3	*Lauha Bhasma*-A	89.04	83.06	0.02	3.38	1.03
4	*Lauha Bhasma* - B	98.97	87.56	0.01	4.09	0.96

Table 3 indicates EDS (Energy dispersive X ray spectroscopy) analysis of the samples. *(Ashodhit,Shodhit and Lauha Bhasma –A and B)*

Table 3- EDS (Energy-dispersive X-ray spectroscopy) analysis

Sr. No	Parameter	*Ashodhit Lauha*	*Shodhita Lauha*	*Lauha bhasma –A*	*Lauha bhasma –B*
1	*Fe*	99.04 %	99.18 %	36.8 %	57.04 %
2	*O*	29.58 %	33.64 %	25.97 %	39.83 %
3	*C*	1.47 %	2.74%	4.94 %	3.14 %
4	*S*	0.04 %	ND	3.24 %	1.95 %
5	*Mg*	0.52 %	0.34 %	0.36%	0.27 %
6	*K*	0.67 %	0.54 %	10.7%	12.65 %
7	*Ca*	0.04 %	0.02 %	1.36 %	0.75 %
8	*Si*	0.17 %	0.15 %	2.74 %	1.97 %
9	*Al*	0.15 %	ND	1.86 %	2.31 %
10	*Na*	0.48 %	ND	0.67 %	0.53 %
11	*Hg*	ND	ND	ND	ND
12	*Mn*	0.06 %	0.02 %	1.25 %	0.96 %

Discussion-

Shodhana (purification) of raw *Lauha,* is the importance step to eliminate impurities and prepare the metal for medicinal use as the transformation of *Lauha* from its original metallic form into a coarse powder is indicative of changes in its physical properties.

The *Drava dravyas* employed for the *Samanya shodhana* of metals exhibit characteristics *Ushna veerya,Teekshna,* and *Sukshma guna,* along with the property of *Bhedana* quality. Most of these substances tend to be either acidic or alkaline in nature and function as chelating agents. Therefore, the *Nirvapa* method for the purification of *Lauha* effectively removes soluble, volatile, washable, and thermo-stable impurities from the metal, rendering it more fragile in its purified state. A reduction in the weight of *Lauha* post *Shodhana* in each liquid medium was commonly observed, except in the case of *Til tail.* Upon undergoing *Vishesha Shodhana,* further reduction in particle size occurs in *Lauha* due to the presence of tannins and the acidic nature of *Triphala kwatha.* Notably, *Triphala* contains ascorbic acid, which enhances the bioavailability of Iron. Both *Samanya* and *Vishesha shodhana*, disrupts the compression-tension equilibrium, resulting in increased brittleness, reduced hardness, and ultimately a decrease in particle size.[10]

During the process of *Marana,* metals undergo a transformation into their mixed oxides. The zero valancy state of the metal is elevated to higher oxidation states through this process. This conversion not only eliminates the toxic nature of resulting metal oxides but also infuses them with medicinal properties

For the *Marana* of *Lauha,* the *Trividha Paka* method (Method A) and *Hingula marit Lauha Bhasma* (Method B), was adopted. Method A required 30 *Puta* while Method B required 7 Puta cycles for proper *Bhasma* formation. Yield was higher in *Lauha Bhasma* B. [11]

In *Trividha Putapaka*,the role of sunlight during *Bhanupaka* serves a specific purpose .The presence of ultraviolet radiation in sunlight facilitates the reduction of the oxidation state of Fe, which, in conjunction with the Vitamin C found in *Triphala* decoction, enhances its bioavailability. In *Trividha Paka*, *Triphala* plays a pivotal role as an organic medium in the conversion of metallic iron (*Lauha)* into a herbomineral complex. Its primary constituents include tannins, gallic acid, ascorbic acid (Vitamin C), and phenolics. *Triphala*'s mild laxative properties counterbalance iron's constipating effects.[12]

In Physiochemical Analysis, the total ash content provides insight into the mineral content of the samples. *Shodhita Lauha* has a high total ash content (47.36%) compared to the *Ashodhit Lauha (*102.21%), demonstrating the removal of organic matter during *Shodhana.Shodhita Lauha* contains 33.52% acid-insoluble extractive, indicating the presence of certain minerals or impurities. In *Lauha Bhasma* A, the water-soluble ash content is exceptionally low (0.02%), suggesting thorough purification. *Lauha Bhasma* B also has low water-soluble ash (0.01%). Both *Lauha Bhasma* A and B exhibits a low pH value, indicating acidity. The pH values are lower for *Lauha Bhasma* A (3.38) compared to *Lauha Bhasma* B (4.09). *Lauha Bhasma* A has a higher LOD (1.03%) compared to *Lauha Bhasma* B (0.96%), suggesting slight variations in moisture content.

Energy-Dispersive X-ray Spectroscopy analysis shows the carbon content exhibited an increase with *Shodhana* process. Absence of detectable sulphur following the *Samanya Shodhana,* possibly due to the repeated heating procedures leads to sulphur oxidation. Meanwhile, trace elements like silica and manganese persisted even after the *Vishesha Shodhana,* albeit with a gradual reduction in their percentage. Notably, the percentage of iron remained nearly constant throughout the entire *Shodhana* procedures. EDS Analysis results show the presence of iron (Fe) in both *Lauha Bhasma* A and B, although the concentration varies significantly between the two. *Lauha Bhasma* B contains a higher iron content (57.04%) compared to *Lauha Bhasma* A (36.8%). This difference underscores how variations in the Marana process can affect the elemental composition of the final product. It also detected some minor elements this may be because of their addition during the *Puta* process; these minor elements undergo oxidation and persist as integral components within the final *Bhasma* product. Notably though *Hingula* was a part of ingredients for Marana of Method B, Hg was not detected in elemental analysis.

Conclusion-

Elemental and physiochemical analyses play a crucial role in assessing the quality and composition of *Lauha Bhasma.* These analyses reveal variations in iron content, ash content, water solubility, pH, and moisture content between the two samples, *Lauha Bhasma* A and *Lauha Bhasma* B. These differences can be attributed to variations in the Marana process and underscore the importance of precise preparation techniques in *Ayurvedic* medicine.

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