ISSN:3139-0935(Online)

Intemed: International Journal of Research in Integrated Medical Sciences (IJ-RIMS)

Journal homepage: www.intemed.in

Vol.: 2 | Issue: 8 | August 2026

Article History: Received: 01-08-2026 Accepted: 5-08-2026 Published:30-08-2026

Review Article

Transformation of Metals during *Shodhana* and *Marana*: Evidence from XRD, XPS, Raman Spectroscopy and Electron Microscopy

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Abstract

The preparation of metal- and mineral-based medicines is a distinctive part of *Rasashastra*. Raw materials are not considered equivalent to the finished *Bhasma*. They undergo procedures such as *Shodhana*, *Bhavana* and *Marana*, which may include heating, quenching, grinding, treatment with liquid media and repeated calcination. This narrative review examines the changes produced by these processes using X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Studies of *Tamra Bhasma*, *Lauha Bhasma*, *Swarna Makshika Bhasma*, *Naga Bhasma*, *Abhraka Bhasma* and *Swarna Bhasma* show the formation of new crystalline phases, altered oxidation states, smaller crystallites and clear changes in surface morphology. The evidence also shows that crystallite size, primary particle size and agglomerate size are different measurements and should not be used interchangeably. Modern analytical methods can strengthen identity testing and batch standardization, but they do not replace classical *Bhasma Pariksha*. Physicochemical transformation or a nanoscale structure alone does not prove clinical efficacy or safety. More reliable conclusions will require stage-wise sampling, complete heating records, validated analytical methods, toxicological assessment and appropriately designed clinical studies.

Keywords: *Rasashastra; Bhasma; X-ray diffraction; X-ray photoelectron spectroscopy; electron microscopy*

Introduction

Rasashastra is the branch of Ayurveda concerned with the pharmaceutical processing and therapeutic use of metals, minerals and related substances. The raw material is not treated as interchangeable with the finished medicine. Its identity is shaped by a sequence of operations performed before it is considered suitable for use [1].

Classical texts describe material selection, *Shodhana*, *Bhavana*, *Marana*, measured heating through *Puta*, and tests used to assess a completed *Bhasma* [2-4]. Official formularies and pharmacopoeial standards add requirements for identity and quality [5,6]. These procedures vary by substance, but they share an important pharmaceutical idea: the final product should be judged as a transformed material, not simply as a powdered version of the starting metal.

Reviews of *Rasaushadhi* and classical metal pharmacology make the same distinction [7,8].

This distinction also makes chemical sense. Copper metal, copper oxide and copper sulfide contain copper, but their crystal structure, oxidation state, solubility and surface behaviour differ. Elemental assays are therefore useful but incomplete. Studies of *Bhasma* composition have shown why elemental content should be interpreted together with chemical form, phase and morphology [9-11].

Modern materials science offers tools for this purpose. XRD identifies crystalline phases; XPS examines surface elements and oxidation states; Raman spectroscopy provides a vibrational fingerprint; and SEM and TEM show morphology at different scales. This review asks what metals and minerals become after repeated Ayurvedic processing, how confidently that change can be measured, and what those measurements cannot establish.

Literature Search Strategy

This narrative review considered classical descriptions of *Shodhana*, *Marana*, *Bhasma Nirmana* and *Bhasma Pariksha*, together with official Ayurvedic formularies and pharmacopoeial standards. Modern literature was searched in PubMed, Scopus, Web of Science and Google Scholar using combinations of "Bhasma characterization," "*Shodhana*," "*Marana*," "XRD," "XPS," "Raman spectroscopy," "SEM," "TEM," and the names of individual preparations. English-language experimental studies and relevant reviews were included when they described the preparation or characterized raw, purified, intermediate or final samples. Greater weight was given to stage-wise studies because they show when a change occurred. The review is interpretive rather than quantitative because differences in raw materials, media, heating systems and analytical methods prevent meaningful pooling.

Pharmaceutical Basis of Transformation

Shodhana as a preparatory modification

Shodhana is often translated as purification, but that word can be too narrow. Depending on the material, the procedure may involve repeated heating and quenching in *Tila Taila*, *Takra*, *Gomutra*, *Kanji*, *Kulattha Kwatha* or another prescribed medium. Repeated heating and rapid cooling create thermal stress, which can produce cracks, increase brittleness and make later grinding easier. Processing liquids may also react with the surface or remove selected constituents. The result depends on the material, temperature, duration, medium and number of cycles; *Shodhana* is therefore better understood as a controlled preparatory modification than as washing alone.

Marana as a thermochemical and mechanochemical process

Marana usually combines fine grinding, wet trituration through *Bhavana*, pellet formation and repeated heating. Grinding exposes fresh surfaces. *Bhavana* brings the inorganic material into close contact with a chosen liquid medium. During drying and heating, oxidation, sulfidation, decomposition, crystallization and other solid-state reactions may occur. The repeated cycle of heating, cooling and regrinding can move the material progressively away from the physicochemical identity of the original metal. The pathway is substance-specific and must be demonstrated rather than assumed.

Analytical Methods

No single instrument can fully describe a complex *Bhasma*. Each method answers a different question, as summarized in Table 1. Strong characterization therefore combines phase, surface, morphology and composition data from the same documented batch.

Table 1: Analytical methods used to examine transformation during *Shodhana* and *Marana*

Technique	Main information	Practical relevance
XRD	Crystalline phases and crystallite dimensions	Tracks disappearance and formation of crystalline compounds
XPS	Surface elements and oxidation states	Distinguishes metallic, oxidized and mixed surface states
Raman spectroscopy	Vibrational fingerprint	Supports identification of oxides, sulfides and related phases
SEM	Surface shape, texture and agglomeration	Shows changes in particle architecture and porosity

TEM	Nanoscale structures and lattice information	Separates small primary structures from larger agglomerates
EDX/EDAX	Elements present in selected areas	Links morphology with local elemental composition

XRD is especially useful because a crystalline phase produces a characteristic diffraction pattern. It can show whether a starting metal remains, whether an oxide or sulfide has formed, and whether the pattern changes with successive *Puta*. Crystallite dimensions estimated from peak broadening are not the same as whole-particle size. A particle seen by microscopy may contain several crystallites or may be part of a larger agglomerate.

XPS adds surface-sensitive information. Binding energies help distinguish oxidation states and surface-associated elements, but the result describes only the outer few nanometres and may not represent the bulk. Raman spectra can help separate structurally related oxides or sulfides and can examine small areas with little sample preparation. SEM is best for broad surface morphology, while TEM provides much finer spatial information. Both are vulnerable to selection bias if only a few visually striking fields are reported.

Evidence from Representative Preparations

Tamra Bhasma

Tamra Bhasma is a clear example of oxidation during processing. Wadekar and colleagues used XRD, XPS and other methods and reported predominantly cupric oxide, with XPS supporting copper in the +2 state [12]. The tested product was therefore chemically different from powdered metallic copper. Earlier experimental studies of *Tamra Bhasma* addressed lipid peroxidation and hepatic effects [13,14], but those biological findings should be interpreted separately from material identity and cannot be generalized to all preparations.

Lauha and Mandura Bhasma

Work on *Lauha Bhasma* shows that purification media and later heating both contribute to transformation. A stage-wise study reported measurable changes during the different purification steps [15], while research on *Bhanupaka* described the chemical participation of plant material in iron processing [16]. Toxicity testing of *Lauha* and *Mandura Bhasma* and physicochemical characterization of *Mandura Bhasma* add useful information, but batch composition, dose and study design remain important limits [17,18]. More recent stage-wise work has also shown changes in iron oxidation, morphology and trace constituents across the full preparation sequence [19].

Swarna Makshika Bhasma

Swarna Makshika is informative because the starting material is commonly related to chalcopyrite rather than a pure metal. In one study, raw material was mainly identified as CuFeS2, whereas the finished *Bhasma* contained iron oxide, iron sulfide, copper sulfide and silica [20]. TEM and EDAX work found pronounced changes from the raw mineral to smaller processed structures [21]. A study comparing conventional *Shodhana* and *Marana* further showed that measured properties change across the process [22]. Together, these findings support decomposition and rearrangement of the parent mineral, while also showing that the final phase mixture depends on the route.

Naga Bhasma

A characterization study of *Naga Bhasma* identified crystalline lead sulfide and reported nanoscale crystallites within larger agglomerated particles; XPS supported lead in the +2 state [23]. Other pharmaceutical and review papers describe traditional preparation and its quality-control challenges [24,25]. A later stage-wise study followed samples through repeated *Puta* and reported progressive changes toward a lead-sulfide-containing final material [26]. These findings document chemical conversion, but they do not remove the need for dose-specific toxicology and clinical caution.

Abhraka Bhasma

Two preparation routes for *Abhraka Bhasma* were compared using XRD, Raman spectroscopy, SEM, TEM and other methods. Both retained a mica-related framework, while crystallite dimensions, morphology and particle distribution differed between the methods [27]. A separate study of *Krishna Vajra Abhraka Bhasma* reported square-shaped particles, oxide phases and organic functional groups associated with processing materials [28]. Earlier analytical work also reported method-dependent physicochemical features [29]. These studies show why one batch cannot stand for every classical variant.

Swarna and Rajata Bhasma

Gold behaves differently from metals that oxidize readily. Characterization of *Swarna Bhasma* has described metallic gold within agglomerated particulate material [30]. Comparative cell studies found that incinerated gold particles and chemically synthesized gold particles did not behave identically during cellular entry [31]. Later work reported nanostructured gold in a carefully prepared product [32]. A study of *Rajata Bhasma* combined material characterization with zebrafish and antimicrobial testing [33]. These findings support batch-specific identity and mechanistic research; they are not direct proof of clinical efficacy.

The main findings and their limits are compared in Table 2. The table separates what the instruments measured from what still requires biological evidence.

Table 2: Representative analytical findings after Ayurvedic processing

Preparation	Main observation	Interpretive limit
Tamra Bhasma	Predominantly cupric oxide and Cu2+ in the studied batch [12]	Total copper does not define chemical form
Lauha Bhasma	Step-wise surface and chemical changes; herbal media participated in iron chemistry [15,16,19]	Outcome depends on the exact sequence and media
Swarna Makshika Bhasma	Parent mineral changed to a mixed-phase product with smaller structures [20-22]	Phase composition can vary with the preparation route
Naga Bhasma	Lead-sulfide-containing phases and nanoscale crystallites were reported [23,26]	Chemical conversion does not replace toxicology
Abhraka Bhasma	Mica-related and oxide phases with method-dependent morphology [27-29]	One batch cannot represent every classical variant
Swarna Bhasma	Metallic nanostructured gold within agglomerated material [30-32]	Nanoscale structure alone does not prove benefit

Discussion

An integrated interpretation

Across the literature, transformation occurs at several connected levels. Physical change includes fragmentation, porosity and altered morphology. Chemical change includes oxidation, sulfidation and the formation of new compounds. Structural change appears when the original diffraction pattern weakens or disappears and new phases develop. Surface change appears as altered oxidation states or surface-associated elements. Compositional change may also reflect liquid media, earthen containers or grinding equipment.

These levels should not be combined into a single claim. A chemically transformed particle is not automatically nanoscale, and a nanoscale crystallite may remain trapped in a much larger agglomerate. Reviews that describe *Bhasma* as ancient nanomedicine have helped draw attention to small particle dimensions [34,35], but the term should be used cautiously. A further discussion of Ayurveda in relation to nanomedicine similarly emphasizes the need for careful characterization [36]. Size, distribution and behaviour must be measured, not inferred from one image or from a classical fineness test.

Classical Bhasma Pariksha and instrumental analysis

Classical *Bhasma Pariksha* developed from practical pharmaceutical experience. Tests such as *Nischandratva*, *Rekhapurnatva*, *Varitaratva*, *Niruttha* and *Apunarbhava* examine features expected in a finished preparation. Modern instruments answer more specific questions, but the two systems should not be described as exact equivalents. Their complementary roles are shown in Table 3.

Table 3: Complementary roles of classical and instrumental assessment

Classical test	Practical meaning	Instrumental support
Nischandratva	Absence of visible metallic lustre	XRD and XPS can investigate residual metallic

		phase or surface state
Rekhapurnatva	Powder enters the fine lines of the fingers	SEM, TEM and size-distribution methods define morphology and dimensions
Varitaratva	A fine layer floats on still water	Density, surface area and wetting studies describe related physical behaviour
Niruttha	No alloying with silver under the prescribed test	Phase analysis before and after testing can examine stability
Apunarbhava	No return to the original metal under the prescribed procedure	XRD, microscopy and thermal analysis can document phase stability

Standardization, safety and limits of interpretation

The practical value of analytical science lies in defining identity, detecting variation and linking a finished product to a documented process. Standardization should include raw-material authentication, exact processing media, temperature-time records, number of cycles, yield, classical tests and a selected analytical panel. Standardization reviews have repeatedly noted the need to combine traditional quality tests with reproducible instrumental criteria[37].

Batch comparison is equally important. A study of five commercial *Tamra Bhasma* products found differences in chemical phase, particle characteristics and pharmacokinetic measures in rats, showing that the name of a preparation alone does not guarantee physicochemical equivalence [38].

Safety is a separate question. Conversion from a metal to an oxide or sulfide may change solubility and biological behaviour, but the direction and size of that change cannot be assumed. Likewise, a nanoscale structure may affect absorption and cellular interaction, yet it does not automatically mean greater safety or efficacy. Blood-compatibility work on *Swarna Bhasma* is useful as a specific preclinical observation [39], while broader safety claims still require impurity testing, dissolution or bio accessibility studies, dose-relevant toxicology and appropriately designed clinical research.

Research Gaps and Future Directions

Many studies analyze only the final *Bhasma*. This confirms an endpoint but does not show the pathway by which it formed. Future work should collect comparable samples from the raw material, after each major *Shodhana* step, after *Bhavana*, after selected *Puta* cycles and from the final product. Every sample should be examined with the same validated analytical methods. A recent bibliometric analysis likewise found that much of the published *Bhasma* literature remains preclinical and that standardization, toxicology, pharmacokinetics and clinical validation remain underrepresented [40].

Heating must also be recorded in measurable terms. A classical term such as *Gajaputa* carries pharmaceutical meaning, but reproducibility additionally requires peak temperature, heating rate, time above selected temperatures, cooling profile and atmosphere. Table 4 presents a practical minimum reporting set.

Table 4: Minimum reporting set for stage-wise *Bhasma* characterization

Domain	Information to report	Reason
Raw material	Source, authentication, composition and impurity profile	Defines the true starting point
Processing	Media, quantities, duration, grinding and container details	Allows replication and source attribution
Heating	System, peak temperature, heating rate, holding time and cooling curve	Links phase change with thermal exposure
Sampling	Raw, purified, intermediate cycles and final product	Shows the transformation pathway
XRD/ XPS	Reference files, acquisition settings, fitting methods and uncertainty	Improves confidence in phase and state assignment
Microscopy	Preparation, magnification, fields examined and size-count method	Reduces image-selection bias
Biological work	Dose, dissolution or bioaccessibility, toxicology and outcomes	Prevents identity from being

		mistaken for clinical proof

Future studies should also publish complete analytical data, use appropriate controls and distinguish crystallite size from particle and agglomerate size. When a processing medium is proposed as the source of an element or functional group, medium-only and equipment controls are needed. Material characterization, toxicology and clinical research should remain linked but methodologically separate.

Conclusions

Modern analytical studies show that *Shodhana* and *Marana* can substantially change the physical and chemical identity of metals and minerals used in *Rasashastra*. XRD demonstrates the disappearance and formation of crystalline phases; XPS clarifies surface oxidation states; Raman spectroscopy adds a vibrational fingerprint; and SEM and TEM reveal changes in morphology, crystallites and agglomeration.

The evidence is strongest when several techniques examine stage-wise samples from the same documented batch. *Tamra Bhasma* illustrates oxidation, *Naga Bhasma* illustrates sulfidation, and *Swarna Makshika Bhasma* shows decomposition and rearrangement of a complex mineral. Studies of *Lauha*, *Abhraka* and *Swarna Bhasma* further show that the processing media and preparation route can influence the final structure.

Classical *Bhasma Pariksha* and instrumental analysis should be used as complementary layers of quality assessment. Neither chemical transformation nor nanoscale structure alone proves safety or therapeutic efficacy. Stage-wise characterization, transparent reporting, toxicological assessment and clinical investigation are all needed before broader conclusions can be made.

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