Research Article

Pharmaceutico-Analytical Standardization of Mandura Bhasma: Correlating Classical Samskaras With XRD Phase Transformation and SEM-EDX Characterization

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Abstract

Horizontal periodontal defects are among the most prevalent forms of alveolar bone loss, yet they remain difficult to regenerate because of limited bony support and reduced regenerative potential. This study aimed to compare the clinical and radiographic results of intraoral autogenous bone graft (ABG) and combined use of demineralized bone matrix (DMBM) and injectable platelet-rich fibrin (I-PRF) for horizontal periodontal defects. Methods: The method used was randomized, single blind, and controlled clinical trial involving 40 patients with chronic periodontitis, who were divided into two groups. A randomized single blind controlled clinical trial was conducted involving 40 patients with chronic periodontitis, divided into two groups. Group A was treated with ABG, and Group B with DMBM and I-PRF. At baseline, 3-months and 6-months, clinical parameters including periodontal probing depth (PPD), relative clinical attachment level (RCAL), plaque index (PI), gingival index (GI), bleeding on probing (BOP) and residual bone level (RBL) were assessed. Repeated-measures ANOVA, paired t-test and unpaired t-test were used to analyze the data. Results: Statistically significant improvement was seen in all six parameters (PPD, RCAL, PI, GI and RBL) at the six months follow up period in both groups ($p < 0.001$). The intergroup comparisons, however, did not show any statistically significant difference in any clinical or radiographic parameter ($p > 0.05$). Conclusion: ABG and DMBM in combination with I-PRF are effective regeneration strategies in horizontal periodontal defects. The DMBM + I-PRF combination is a minimally invasive option that has similar clinical results to the gold standard autogenous bone graft.

Keywords: Mandura Bhasma; Rasashastra; XRD; SEM-EDX; Magnetite; Standardisation

Introduction

Rasashastra is a specialized branch of Ayurveda that deals with the processing of metals and minerals into therapeutically potent and biologically acceptable forms. Among these, Mandura (iron slag) occupies a significant position owing to its therapeutic action in conditions ranging from Grahani vikara to Panduroga [1]. Classical Ayurvedic texts describe a systematic biotransformation of Mandura into Mandura Bhasma through a series of samskaras, namely Shodhana (purification), Bhavana (trituration), and Marana (incineration) carried out by repeated Puta.

These processes are not merely physical modifications but involve profound physicochemical transformations, including structural breakdown, detoxification, and enhancement of bioavailability. Repeated thermal processing, quenching, and grinding break down the natural crystalline matrix of the mineral, transforming it into a stable, fine-particle calx optimized for bioavailability. Modern investigations suggest that these transformations may yield iron-oxide based particles of micro to nano dimensions, which could account for the improved absorption and reduced toxicity attributed to the finished Bhasma.[2]

In contemporary research, there is a growing need to scientifically validate these traditional processes using modern analytical tools. Establishing correlations between classical Ayurvedic parameters and instrumental findings is essential for standardization, reproducibility, and global acceptance. Advanced techniques such as XRD and SEM-EDAX can elucidate the structural, phase, and elemental changes occurring across the processing stages, thereby providing an objective basis for quality control. Therefore, the present study aims to comprehensively evaluate Mandura Bhasma through both classical Ayurvedic tests and advanced analytical techniques, with the objective of establishing scientific standardization parameters for this classical formulation.[3]

Materials And Methods

The present study was conducted as an experimental pharmaceutico-analytical investigation involving classical preparation followed by detailed analytical evaluation.

The process began with Shodhana, in which raw Mandura was subjected to repeated heating and quenching (Nirvapa) cycles in

a suitable liquid medium, commonly Gomutra[4]. This step plays a crucial role in removing physical and chemical impurities while simultaneously inducing brittleness in the mineral. The sudden temperature variation creates microfractures within the layered silicate structure, facilitating its subsequent breakdown. As a result of this process, the Mandura became progressively more brittle, gradually transitioning from a solid mass to a coarse powdered form.

In the context of Rasashastra, Marana refers to the process of incineration or calcination, in which the metallic, non-metallic, and mineral nature of a substance is destroyed and converted into a fine, bioavailable Bhasma (calx) suitable for medicinal use. For the Marana of Mandura, the following techniques are generally adopted. Since Mandura is an iron-based substance, the same methods and drugs employed for the processing of Lauha may be used; accordingly, the drugs of the Lauha Maraka Gana are applied for Mandura Marana. Triphala Kwatha is the

most commonly used liquid medium for Bhavana (trituration), and Gajaputa is generally employed as the heating grade (Puta) for Mandura[5,6].

After each Puta, the material was examined for the classical Bhasma Pariksha (tests of completion) such as Varitara (floating on water), Rekhapurnata (filling the lines of the fingers), and Nishchandratva (absence of metallic lustre), and the Puta cycle was repeated for 13 times until these parameters were satisfied. Marana is responsible for detoxification, particle size reduction to micro/nano scale, and structural transformation into a biologically compatible form.

Mandura Shodhana

Bhavana of Mandura

Marana of Mandura

Mandura Bhasma

Rekhapurnatva

Varitaratva

PHYSICO-CHEMICAL PARAMETERS Classical Analytical parameters

Organoleptic parameters:

The prepared Mandura Bhasma was observed as a fine, smooth, brown powder, devoid of any odour or taste. The smooth texture indicates effective particle size reduction and proper incineration.

Nishchandratva Pariksha:

After completing the Marana process, the pellets of Mandura were examined in sunlight. A small amount of the resulting Bhasma was then taken on the palm and observed again under sunlight to check for the presence of any lustrous particles.[7]

Rekhapurnatva Pariksha:

A pinch of Bhasma is rubbed between the index finger and thumb. If the Bhasma enters and becomes embedded in the furrows of the skin, it indicates Rekhapurnatvam. This signifies that the Bhasma is sufficiently fine and meets the required standards.[8]

Varitaratva Pariksha:

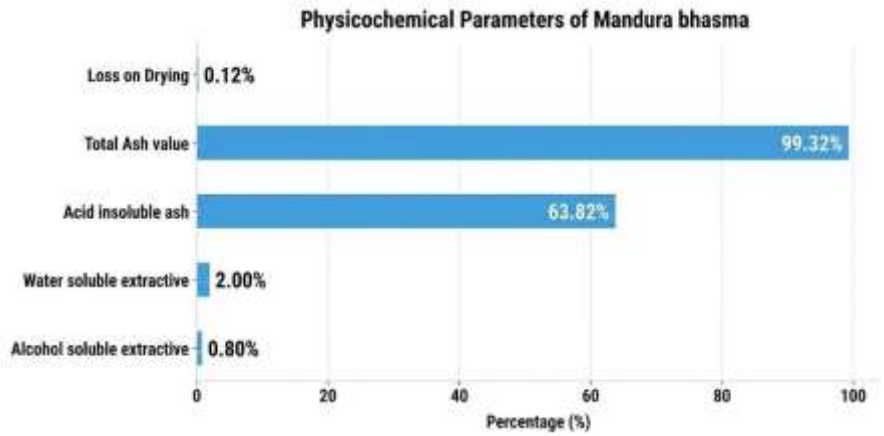
A pinch of the Bhasma, after undergoing the process of incineration and fine trituration, is gently placed on the surface of still water in a glass. The Bhasma should float and should not sink. This indicates the Varitara property of the Bhasma. At this stage, the incinerated metallic– mineral Bhasma becomes so fine that it does not break the surface tension of the water.[9]

Physico-chemical Analysis

The physico-chemical parameters revealed that the formulation had very low moisture content, indicating high stability and reduced risk of degradation. The total ash value was significantly high, confirming complete inorganic conversion of the material. Extractive values were minimal, suggesting absence of residual organic impurities.

Table no. 1 – Physico-chemical parameters of Mandura Bhasma

S.no.	Physico-chemical parameters	Mandura bhasma
1	Loss on Drying	0.12%
2	Total Ash value	99.32%
3	Acid insoluble ash	63.82%
4	Water soluble extractive	2%
5	Alcohol soluble extractive	0.8%



The graphical representation highlights the dominance of ash value, supporting complete Marana and inorganic nature.

ANALYTICAL STUDY SEM-EDX Analysis

SEM analysis revealed a uniform and markedly reduced particle morphology. EDX analysis showed a progressive reduction in the silicate matrix elements, with silicon decreasing from 17.29% to 14.92% and aluminium from 12.43% to 8.41% across the processing stages. Concurrently, trace essential elements were incorporated from the processing media, magnesium and calcium appearing in the incinerated samples while being absent in raw Mandura. The iron content was largely retained (27.86% → 23.56%), the relative shift being attributable to the breakdown of the aluminosilicate matrix and the introduction of mediaderived elements. These changes confirm effective purification, matrix disintegration, and enrichment of the Bhasma with biologically essential trace elements.

Table no. 2 - Particle size of various stage of Mandura

S.N.	Type of Mandura	Length
1.	RM	90.989 μm
2.	MB 1	0.468 μm
3.	MB 13	261.476 nm

RM – Raw Mandura
MB1 – Mandura Bhasma after 1 puta
MB13 – Mandura Bhasma after 13 puta

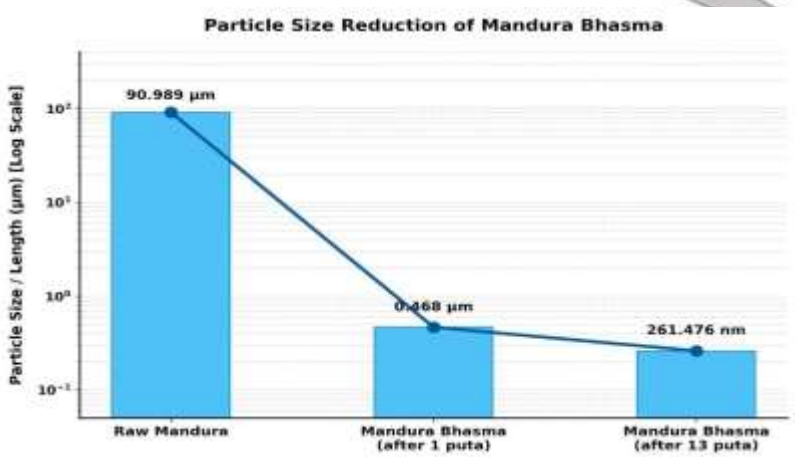


Table No. 3 - Elemental composition in various stage of Mandura

S.N.	Element	Weight % RM	Weight % MB1	Weight % MB13
1	O K	42.42	40.41	42.30
2	Al K	12.43	10.13	8.41
3	Si K	17.29	16.35	14.92
4	Fe K	27.86	23.24	23.56
5	Mg K	-	2.52	2.82
6	K K	-	2.23	-
7	Ca K	-	5.01	4.65
8	C K	-	-	3.33

Table no. 4 – XRD of various stages of Mandura

Parameters	RM	ShM	MB1	MB13
Reference code	96-901-0048	96-901-0048	96-900-6922	96-900-4157
Compoun d name	Stilpnomelan e	Stilpnomelane(wi th incipient fayalite)	Magnetite	Magnetite
Chemical formula	$Fe_{48}Si_{72}O_{216}$	$Fe_{48}Si_{72}O_{216}$	$Fe_{23.28}Si_{0.72}O_{32}$	$Fe_{16.80}Mn_{0.16}V_{0.08}Ti_{5.92}Ca_{0.08}Si_{0.08}Al_{0.40}Mg_{0.48}O_{32}$

Crystal system	Anorthic (Triclinic)	Anorthic (Triclinic)	Cubic	Cubic
Cell volume (10^6 pm^3)	5199.11	5199.11	591.01	614.99
RIR	8.76	8.76	5.15	4.71

The XRD pattern of Raw Mandura (RM) revealed peaks corresponding to Stilpnomelane with an Anorthic (Triclinic) crystalline phase. Shodhita Mandura (ShM) retained the dominant Stilpnomelane matrix with an Anorthic (Triclinic) crystalline phase; however, two new diagnostic reflections appeared at 24.808° (d = 3.586 Å) and 28.078° (d = 3.175 Å), with intensities of 168.9 and 170.1 respectively, corresponding to fayalite (Fe_2SiO_4). As these reflections are completely absent in raw Mandura, their emergence indicates the onset of a thermochemical transformation during Shodhana, without a wholesale change in the primary crystalline matrix.

The XRD pattern of Mandura Bhasma after the 1st puta (MB1) demonstrated peaks of Magnetite with a Cubic crystalline phase. The XRD pattern of Mandura Bhasma after the 13th puta (MB13) likewise showed peaks of Magnetite with a Cubic crystalline phase, confirming phase stabilization following repeated Marana.

The cell volume was observed to decreases progressively from RM to MB13, with slight increase noted between MB1 and MB13. Similarly, the RIR value showed a progressive decrease from RM to MB13. These finding conclusively support the XRD-based theory of particle size reduction, which was also in the particle size data presented earlier.

This transition from a triclinic silicate (Stilpnomelane) to a cubic iron-oxide phase (Magnetite) indicates a complete mineralogical reorganization of the material during marana. The finished Bhasma remains crystalline, as confirmed by well-defined magnetite reflections and a discrete cubic unit-cell volume, however the broadening of diffraction peaks reflects a marked reduction in crystallite and particle size to nanoscale rather than any loss of crystallinity. This size reduction enhances surface area, solubility, and bioavailability. The progressive decrease in particle size from raw mandura to the finished Mandura Bhasma confirms the effectiveness of sequential processing stages.

Discussion

In the present study, Shodhana (purification) of Mandura was carried out by subjecting 1.5 kg of raw (Ashuddha) Mandura to repeated quenching in 21 liters of cow's urine (Gomutra). The final yield obtained post-purification was 1.49 kg, indicating a minor weight loss of 10 gm. This marginal loss can be attributed to the fragmentation of brittle Mandura particles during the thermal shocking process, causing fine particulates to mix with the Gomutra and subsequently wash away during filtration.

X-ray diffraction (XRD) analysis after Shodhana revealed no major alteration in the primary crystalline matrix. However, the XRD profile of Shodhita Mandura distinctly unveiled two sharp, diagnostic reflections at 24.808° (d = 3.586 Å) and 28.078° (d = 3.175 Å) exhibiting strong intensities of 168.9 and 170.1, respectively. These specific peaks correspond directly to fayalite (Fe_2SiO_4), an iron-rich silicate mineral. The appearance of these characteristic fayalite reflections completely absent in raw Mandura indicates the onset of thermochemical transformation induced by Shodhana. While the dominant Stilpnomelane matrix is retained, the emergence of incipient fayalite marks the beginning of the mineralogical reorganization that prepares the raw iron slag for the more extensive phase change achieved during subsequent Marana.

Following purification, 1.48 kg of Shodhita Mandura was processed for Marana (incineration). The liquid medium (Marana Dravya) used was Triphala decoction (Kwatha). Prior to each incineration cycle (Puta), the material was subjected to 7 cycles of levigation (Bhavana), consuming a cumulative total of 15 liters of Triphala Kwatha across 13 sequential Putas. Concluding the incineration stage, 1.43 kg of finished Mandura Bhasma was obtained.

Marana plays a crucial role in converting the mineral material into a stable, non-toxic, and bioavailable form. The high total ash value and exceptionally low moisture content observed in the physicochemical parameters confirm the completeness of the incineration process. Scanning Electron Microscopy energy-dispersive X-ray spectroscopy (SEM-EDX) results validate elemental purification and the successful incorporation of trace essential elements from the processing media. Concurrently, XRD analysis provides direct evidence of structural transformation, Charting the progression from a triclinic stilpnomelane phase in raw mandura to a stable cubic magnetite phase in the final Bhasma. This structural transition is highly significant the conversion to nanocrystalline magnetite evidenced by peak broadening alongside retained crystalline reflections, yields a smaller crystallite size, greater surface area and enhanced interfacial interaction within biological systems, thereby improving solubility and bioavailability.

The graphical representation of the analytical data further strengthens these interpretations by visually demonstrating the

progressive reduction in particle size and elemental evolution across the processing stages. Ultimately, this study successfully establishes a definitive correlation between classical Ayurvedic concepts of Samskara (pharmaceutical processing) and modern scientific principles of materials science and pharmacology.

Molecular Basis of the Physicochemical Transformation

At the molecular level, the observed changes reflect a systematic reorganization of an iron aluminosilicate into an iron-oxide spinel. Raw Mandura corresponds to Stilpnomelane, a hydrous iron–aluminosilicate bearing structural hydroxyl groups and interlayer water. During Shodhana, repeated heating followed by quenching in Gomutra imposes cyclic thermal stress and initiates dehydroxylation, while the reactive constituents of Gomutra (urea, ammonia, and chloride salts) assist in leaching surface impurities. This triggers localized iron-silicate reorganization, evidenced by the emergence of fayalite (Fe_2SiO_4) reflections an iron (II) orthosilicate that signals the onset of iron enrichment within the silicate framework without a wholesale collapse of the parent matrix.

The decisive transformation occurs during Marana. The polyphenolic constituents of Triphala Kwatha (gallic acid, chebulinic and chebulagic acids, and tannins) act as both chelating and reducing agents. With each Bhavana and subsequent Gajaputa, these organic acids chelate iron and, upon pyrolysis within the sealed Puta, generate a carbon-rich, oxygen-limited (reducing) micro-environment. This reducing atmosphere is critical it favours the formation of mixedvalence Magnetite (Fe_3O_4, containing both Fe^{2+} and Fe^{3+}) over the fully oxidized Hematite (αFe_2O_3), which explains why the finished Bhasma stabilizes as cubic Magnetite. Concurrently, the aluminosilicate matrix progressively decomposes, accounting for the EDX-observed decline in silicon and aluminium, while magnesium and calcium derived from the Triphala media are incorporated into the product. The trace carbon (3.33%) detected in MB13 represents residual pyrolytic organic matter from the Triphala Kwatha.

These molecular events are directly mirrored in the physico-chemical data. The very low loss on drying (0.12%) reflects the loss of structural water and hydroxyl groups (dehydroxylation), yielding an essentially anhydrous oxide. The high total ash value (99.32%) confirms complete removal of organic and volatile matter, leaving a purely inorganic phase. The elevated acidinsoluble ash (63.82%) corresponds to residual silica (SiO_2) retained from the parent silicate, which resists acid digestion consistent with the persistent silicon signal in the EDX spectra. Finally, the combined mechanical trituration (Bhavana) and thermal cycling (Puta) drive crystallite and particle-size reduction to the nanoscale, producing the high surface area that underlies the enhanced solubility and bioavailability of the finished Bhasma

Conclusion

The present study successfully establishes a comprehensive pharmaceutico-analytical profile of Mandura Bhasma prepared using classical Ayurvedic methodologies. The resulting formulation satisfies both traditional quality control metrics and modern regulatory standards, undergoing significant structural, mineralogical, and compositional transformations. The integration of classical organoleptic tests with advanced analytical techniques confirms that the traditional pharmaceutical processes specifically Shodhana (purification) and Marana (incineration) induce essential phase transitions, substantial particle size reduction, and an enhancement of potential bioavailability. Ultimately, these findings provide a robust, evidencebased scientific foundation for the standardization of Mandura Bhasma, supporting the global acceptance and therapeutic integration of this traditional herbo-mineral formulation.

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