

Comparative Pharmaceutico-Analytical Evaluation Of Two Shodhana Methods Of Manahshila (Realgar): SEM And XRD Characterization

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ABSTRACT

Background: Manahshila (Realgar) is an arsenic sulphide mineral drug used in Ayurvedic pharmaceuticals only after Shodhana. Because classical texts prescribe more than one processing method, it is relevant to determine whether different Shodhana procedures produce measurable changes in pharmaceutical recovery, surface morphology and crystalline characteristics.

Objective: To comparatively evaluate two classical Shodhana methods of Manahshila using pharmaceutical observations, scanning electron microscopy (SEM) and X-ray diffraction (XRD).

Materials and Methods: Two independent 76 g batches of Raw Manahshila were processed according to Rasa Tarangini. Sample 2A underwent seven Bhavana cycles with fresh Nimbu Swarasa, whereas Sample 2B was immersed in 566 g of pre-prepared Churnodaka for three days. The dried shodhita Manashila Samples 2A & 2B were weighed and characterized by SEM and XRD.

Results: Sample 2A yielded 64 g (84.21%) and Sample 2B yielded 52 g (68.42%). The final Shodhita Manashila Samples 2A & 2B also differed visibly in colour. SEM showed heterogeneous angular particles, adhered fines and agglomerates in both samples, with a relatively denser fine-granular field in Sample 2B. XRD identified the same principal Realgar/arsenic-sulfide phase (PDF 01-071-2434; monoclinic P21/n) in both samples. Selected software-reported crystallite-size estimates averaged 73.93 nm in Sample 2A and 73.20 nm in Sample 2B.

Conclusion: The two Shodhana methods produced different pharmaceutical recovery, colour and micro-morphological appearance while retaining the same principal Realgar crystalline phase. The available data therefore support predominantly physical and surface-level differences rather than a gross bulk-phase transformation.

Keywords: Manahshila; Realgar; Shodhana; Nimbu Swarasa; Churnodaka; SEM; XRD

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INTRODUCTION

Manahshila is an important arsenic sulphide mineral drug of Rasashastra and is commonly correlated with Realgar. Classical pharmaceutical practice requires Shodhana before therapeutic use. Rasa Tarangini describes more than one Shodhana methods for Manahshila, including Churnodaka immersion and repeated Bhavana with Nimbu Swarasa¹

Analytical studies indicate that Shodhana can substantially alter physical characteristics without

necessarily changing the principal arsenic-sulfide phase. Kodlady et al. reported a reduction in particle size after Ardraka Swarasa Shodhana, while both unprocessed and processed Manahshila remained arsenic disulfide on XRD.² A related experimental report also documents seven Bhavana cycles as a practical Manahshila Shodhana procedure³

Reviews of Manashila purification describe the use of plant-derived media, including citrus preparations (

Nimbu swarasa), for Realgar processing.⁴ Modern characterization of Shodhita Manashila increasingly combines SEM and XRD because these techniques provide complementary information on surface morphology and crystalline phase.^{5,6} The present work therefore compared two classically described Shodhana procedures under a common analytical framework. The objectives were to compare: (i) pharmaceutical recovery and visible organoleptic changes; (ii) SEM morphology and agglomeration pattern; (iii) XRD phase identity, peak characteristics and software-reported crystallite-size estimates.

MATERIALS AND METHODS

Study design and sample coding

A comparative pharmaceutico-analytical study was performed on two independently processed 76 g batches of Ashuddha Manahshila. Sample 2A represented Nimbu Swarasa-shodhita Manahshila and Sample 2B represented Churnodaka-shodhita Manahshila. The procedures followed the classical descriptions in Rasa Tarangini, Taranga 11, verse 111 for Nimbu Swarasa Bhavana and verse 109 for Churnodaka immersion.¹

Sample 2A: Nimbu Swarasa Shodhana

Ashuddha Manashila (76 g) was weighed, placed in a Khalva Yantra and reduced to a fine powder. Fresh Nimbu Swarasa (12 mL per cycle) was added in sufficient quantity to uniformly wet the Manashila, followed by continuous Mardana for approximately 2 hours or until the material became sufficiently dry. One Bhavana cycle was performed per day, and the procedure was repeated for seven consecutive days, completing a

total of seven Bhavana cycles. Thus, a total of 84 mL of fresh Nimbu Swarasa was utilized, with an approximate cumulative Mardana duration of 14 hours. After completion of the seventh Bhavana, the Shodhita Manashila was completely dried, powdered and weighed; 64 g was recovered.

Sample 2B: Churnodaka Shodhana

A separate 76 g batch of Ashuddha Manashila was powdered in Khalva Yantra and immersed in 566 g (approximately 566 mL) of pre-prepared Churnodaka for three days.

Analytical characterization

SEM was performed using a JEOL JSM-6390 instrument in secondary electron imaging mode at 20 kV. Micrographs were obtained at $\times 250$, $\times 500$, $\times 1000$, $\times 2000$ and $\times 4000$ magnification and interpreted qualitatively for particle shape, surface texture and agglomeration. XRD was carried out at SAIF Kochi using a coupled TwoTheta/Theta continuous scan with Cu K α radiation (λ 1.54060 Å), 40 kV, 35 mA, 2 θ range 10.000°-79.991° and 0.020° step size. Phase identity, peak position, FWHM, d-spacing and software-reported crystallite-size estimates were taken from the laboratory reports. As one processed batch per method was examined, results were summarized descriptively and no inferential statistics were applied.

Ethical considerations

Not applicable. The work involved pharmaceutical processing and material characterization only; no human participants or experimental animals were involved.

Comparative Pharmaceutico-Analytical Evaluation Of Two Shodhana Methods Of Manahshila (Realgar): Sem And Xrd Characterization



Figure 1. Pharmaceutical processing and comparative appearance of Manahshila. A: Ashuddha Manahshila (76 g); B: fresh Nimbu Swarasa 12ml per batch; C: Nimbu Swarasa-shodhita Manahshila after seven Bhavana cycles (64 g); D: bottle containing Churnodaka (889 g gross; 566 g approximately 566ml net Churnodaka after subtracting 323 g bottle tare); E: transfer/immersion of powdered Manahshila into Churnodaka; F: Churnodaka-shodhita Manahshila after drying (52 g); G: side-by-side appearance of the two final samples.

RESULTS AND DISCUSSION

Pharmaceutical observations

The two procedures produced different process recoveries and visibly different final powders. Sample 2A retained 64 g from 76 g (84.21% recovery; 15.79% loss), whereas Sample 2B retained 52 g (68.42% recovery; 31.58% loss). The Nimbu Swarasa-processed material appeared yellowish-orange, while the Churnodaka-processed material appeared comparatively reddish-orange. These values represent pharmaceutical recovery and not a direct measure of detoxification or arsenic removal.

Parameter	Sample 2A: Nimbu Swarasa	Sample 2B: Churnodaka
Initial weight	76 g	76 g
Final weight	64 g	52 g
Weight loss	12 g	24 g

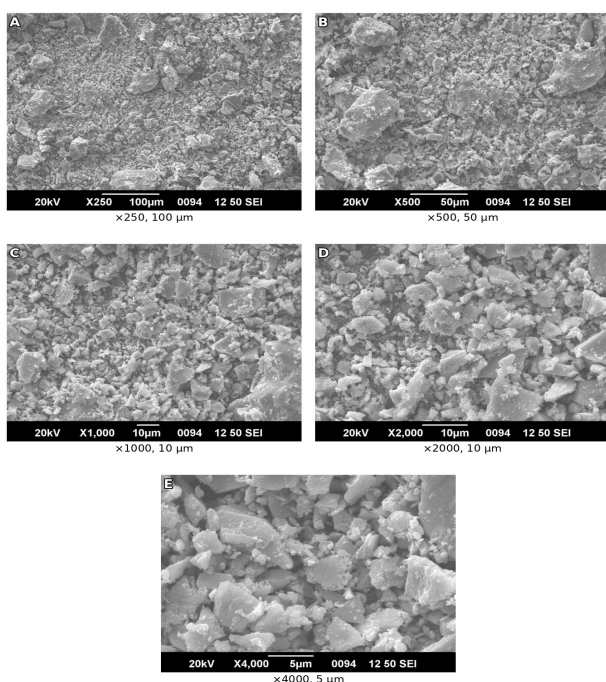
Recovery	84.21%	68.42%
Loss	15.79%	31.58%
Appearance	Yellowish-orange fine powder	Reddish-orange fine powder

SEM morphology

SEM demonstrated heterogeneous particulate fields in both samples, with irregular angular fragments, adhered finer particles, rough/uneven surfaces and agglomeration. Sample 2A showed prominent plate-like or fractured larger particles admixed with smaller fragments. Sample 2B showed a comparatively denser

fine-granular matrix surrounding larger irregular particles in the examined fields. Because calibrated image-based particle-size measurement was not performed, the SEM findings are qualitative and do not establish a statistically smaller particle size for either sample.

A Sample 2A - Nimbu Swarasa



B Sample 2B - Churnodaka

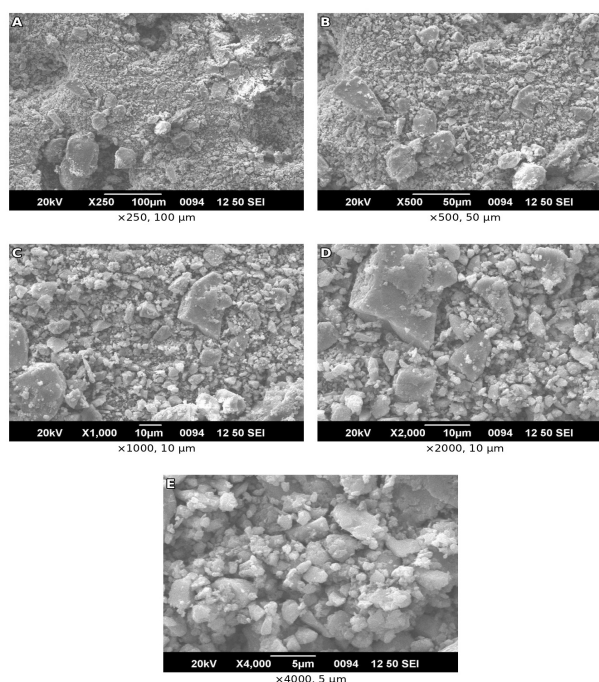


Figure 2. Comparative SEM morphology of processed Manahshila. A: Sample 2A (Nimbu Swarasa-shodhita); B: Sample 2B (Churnodaka-shodhita). Micrographs are shown at $\times 250$, $\times 500$, $\times 1000$, $\times 2000$ and $\times 4000$ magnification with the corresponding scale bars.

XRD characterization

Both samples showed sharp diffraction peaks indicative of crystalline material. The laboratory database match identified the principal phase in both samples as Realgar/arsenic sulfide (PDF 01-071-2434) with monoclinic P21/n (14) symmetry. The matched unit-cell

parameters were $a = 9.325 \text{ \AA}$, $b = 13.571 \text{ \AA}$ and $c = 6.587 \text{ \AA}$. Thus, no gross change in the principal crystalline phase was detected between the two Shodhana procedures.

For four selected peaks reported by the instrument software, 2θ differences between corresponding peaks were only 0.051° - 0.074° . Crystallite-size estimates ranged from 62.0-84.0 nm in Sample 2A and 61.3-80.4 nm in Sample 2B. Their descriptive means were 73.93 and 73.20 nm, respectively. These crystallite estimates should not be equated with SEM particle dimensions because one SEM particle or agglomerate may contain multiple crystallites.

Peak	2A 2θ ($^\circ$)	2A FWHM ($^\circ$)	2A size (nm)	2B 2θ ($^\circ$)	2B FWHM ($^\circ$)	2B size (nm)
1	14.652	0.107	84.0	14.703	0.122	71.3
2	15.424	0.122	71.5	15.493	0.111	80.4

3	16.361	0.113	78.2	16.423	0.111	79.8
4	32.783	0.141	62.0	32.857	0.143	61.3
Mean*	-	-	73.93	-	-	73.20

*Mean of four selected software-reported estimates; not a full-pattern crystallite-size distribution.

A Sample 2A - Nimbu Swarasa

B Sample 2B - Churnodaka

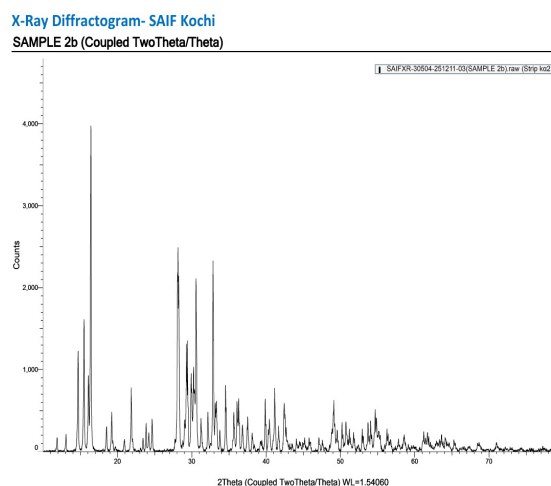
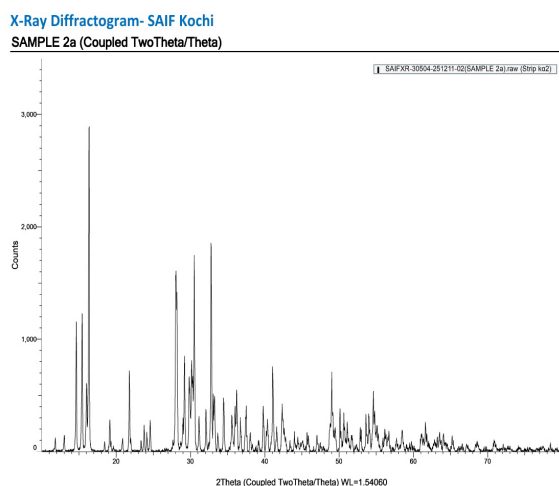


Figure 3. Full X-ray diffractograms of the two processed samples shown at identical horizontal scale. A: Sample 2A (Nimbu Swarasa-shodhita Manahshila); B: Sample 2B (Churnodaka-shodhita Manahshila).

INTEGRATED DISCUSSION OF STUDY OBJECTIVES

The first aspect evaluated was to compare pharmaceutical recovery and visible organoleptic change. The lower recovery after Churnodaka processing (68.42%) than after Nimbu Swarasa Bhavana (84.21%) may likely reflect greater handling and separation loss of fine material during the three-day immersion procedure, including adherence of fines to the vessel or retention in the churnodaka medium; this cannot be interpreted as superior purification by either method. The marked yellowish-orange versus reddish-orange appearance is also important because both samples later showed the same principal XRD phase. This visible differences may also be related to the difference in nature of the two processing media, Nimbu swarasa being acidic & churnodaka being alkaline . However, as the pH of the media was not measured in this study , this remains a possible explanation & not a demonstrated mechanism. A possible explanation is that the two chemically different processing media and procedures may have influenced the Surface

characteristics & Particle aggregation of Manahshila . Repeated wet trituration with Nimbu swarasa may leave a different surface-associated mineral environment & prolonged immersion in Churnodaka (lime water) , followed by drying may have contributed to the observed differences in Surface Morphology & Colour. These are possible mechanisms possibilities not proven chemical transformations; confirmation would require additional techniques such as Raman/FTIR spectroscopy, surface-sensitive elemental analysis or arsenic speciation. Previous Manahshila reports have suggested that Shodhana may alter physical characteristics while the main arsenic-sulfide identity remains detectable.^{2,4} The second objective concerned SEM morphology. The difference in SEM appearance of Sample 2A and Sample 2B may reflect the different Process methods without quantitative image based Particle size, definitive size differences cannot be concluded . The third objectives concerned XRD characterisation. Both samples matched Realgar/arsenic sulfide (PDF 01-071-2434; monoclinic P21/n), and corresponding selected peaks differed by only 0.051°-0.074° in 2θ. Such small shifts can arise from sample packing, preferred orientation, microstrain or minor surface/microstructural differences and, in the absence of a new matched phase, do not demonstrate a bulk structural conversion. The selected crystallite-size estimates were also closely comparable (mean 73.93 nm in 2A versus 73.20 nm in 2B). This phase retention agrees with earlier Manahshila work in which processing

altered particle-size characteristics without changing the main arsenic-sulfide identity, while this phase retention agrees with earlier Manashila reports suggesting that Shodhana may alter Physical characteristics while the main Arsenic Sulphide identity remains detectable. However, other studies on Realgar have reported that different processing conditions may produce additional Arsenic disulphide phases. Therefore, the phase stability observed in the present samples should be considered specific to the processing conditions used in this study^{2,5,6}

LIMITATIONS

Raw Manahshila was not analyzed as a matched SEM/XRD control in the present comparison, and only one processed batch per method was characterized. Quantitative SEM particle-size analysis, replicate batches and separately coded elemental/arsenic-speciation measurements for both 2A and 2B were not available. Therefore, the present study characterizes pharmaceutical and material differences but does not quantify detoxification or arsenic removal.

CONCLUSION

Two classical Shodhana methods of Manahshila produced differences in pharmaceutical recovery, visible colour and qualitative SEM morphology. Nimbu Swarasa Bhavana yielded 84.21% recovery, whereas Churnodaka immersion yielded 68.42%. The Shodhita Manashila samples differed in visible colour & Surface Morphology, but XRD showed the same principal Realgar phase (PDF 01-071-2434; monoclinic P21/n) in both samples, with closely comparable selected crystallite-size estimates. Within the limits of this small comparative study, the findings suggest a method-dependent physical and microstructural effect without evidence of bulk transformation of the main crystalline phase.

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

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