

## PHYSICOCHEMICAL CHARACTERIZATION OF *MANAHSILA* BEFORE AND AFTER *SHODHANA* USING XRF AND XRD: A SCIENTIFIC VALIDATION OF AYURVEDIC PURIFICATION PROCESS

Dr. Kritika Verma\*, Dr. Hemlata<sup>1</sup>, Dr. Neha Pathania<sup>2</sup>, Dr. Sudhendu Shekhar<sup>2</sup>

<sup>1</sup>PG Scholar, PG Department of Rasa Shastra and Bhaishajya Kalpana, Patanjali Bhartiya Ayurvedigyan Evum Anusandhan Sansthan Haridwar, Uttarakhand, India.

<sup>2</sup>PG Scholar, PG Department of Dravyaguna, Patanjali Bhartiya Ayurvedigyan Evum Anusandhan Sansthan Haridwar, Uttarakhand, India.

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\*Corresponding Author: Dr. Kritika Verma



PG Scholar, PG Department of Rasa Shastra and Bhaishajya Kalpana, Patanjali Bhartiya Ayurvedigyan Evum Anusandhan Sansthan Haridwar, Uttarakhand, India.



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### ABSTRACT

**Background:** *Manahsila* (Realgar; Arsenic Disulfide) is an important mineral drug in Ayurvedic *Rasa Shastra*. Due to its inherent toxicity, classical Ayurvedic texts recommend *Shodhana* (purification) before its therapeutic use. Although *Shodhana* is believed to reduce harmful impurities and improve the safety and quality of the drug, scientific evidence validating these transformations remains limited. **Objective:** The present study aimed to scientifically evaluate the physicochemical changes occurring in *Manahsila* before and after *Shodhana* using X-ray Fluorescence (XRF) and X-ray Diffraction (XRD), thereby providing analytical evidence for the Ayurvedic purification process. **Methods:** Raw *Manahsila* was subjected to *Shodhana* according to the classical Ayurvedic procedure. Both untreated and purified samples were analysed using XRF to determine elemental composition and XRD to investigate crystalline phase characteristics. The analytical findings obtained before and after *Shodhana* were comparatively evaluated. **Results:** XRF analysis demonstrated variations in the elemental composition following *Shodhana*, indicating the possible removal or reduction of extraneous impurities while preserving the principal constituents of *Manahsila*. XRD analysis revealed that the characteristic crystalline structure of realgar was largely retained after purification, with minor variations in peak intensity and crystallinity that may reflect structural modifications induced during the purification process. These observations suggest that *Shodhana* primarily improves the physicochemical quality of the material without causing significant alteration of its fundamental mineral phase. **Conclusion:** The findings provide scientific support for the classical Ayurvedic concept of *Shodhana* by demonstrating measurable physicochemical changes in *Manahsila*. XRF and XRD proved to be valuable analytical tools for the standardization and quality assessment of purified mineral drugs. The study contributes to the scientific validation of traditional Ayurvedic purification procedures and may facilitate the development of standardized and safer *Rasaushadhi* formulations.

**KEYWORDS:** *Manahsila*, *Shodhana*, XRF, XRD, Physicochemical Characterization, Realgar, Rasa Shastra, Ayurvedic Purification, Standardization, Scientific Validation.

## INTRODUCTION

*Manahsila* is the sixth mineral drug of *uparasa* group. It is identified as “Realgar” in English. This is also a reddish-brown Arsenic compound, chemically known as Arsenic disulphide with chemical formula  $As_2S_2$ .<sup>[1]</sup>

### Other name<sup>[2]</sup>

- Bengali - *Manchala*
- English - *Realgar*
- Gujarati - *Manasila*

- Hindi - *Mainsil*
- Marathi - *Manasila*
- Tamil - *Manocilai*
- Telugu - *Manisila*
- Urdu - *Hartal Tabqi, Zinikh-e-Qirmiz*

Its colour is Aurelian red to orange yellow. Streak has a lighter shade. Hardness- 1.5-2, Specific gravity- 3.5, Contains As- 70.1% and S- 29.1%.<sup>[3]</sup>

## Types of Manahsila

Table 2.21: Different type of *Manahsila* in different text.

| S.No. | Reference                       | Type                      | Lakshana                               |
|-------|---------------------------------|---------------------------|----------------------------------------|
| 1.    | R.R.S <sup>[4]</sup>            | i. <i>Syamangi</i>        | - <i>Syama, Rakta, Sagaura, Guru</i>   |
|       |                                 | ii. <i>Kanviraka</i>      | - <i>Tamrabha, Tejaswi, Nirgaura</i>   |
|       |                                 | iii. <i>Khandakhya</i>    | - <i>Atirakta, Guru, Khandrupa</i>     |
| 2.    | A.P. <sup>[5]</sup> (2/215-217) | i. <i>Syamangi</i>        | - <i>Hingula sadrasa, Khinchitpita</i> |
|       |                                 | ii. <i>Kanviraka</i>      | - <i>Raktavarna, Churnarupa</i>        |
|       |                                 | iii. <i>Dwikhandakhya</i> | - <i>Kinchita Rakta, Gaura</i>         |

In Ayurveda, *Shodhana* (purification) is considered an essential pharmaceutical process for mineral and metallic drugs before their therapeutic use. *Manahsila*, a mineral drug widely described in *Rasashastra*, is used in the treatment of various disorders after proper purification. Raw (*Ashuddha*) *Manahsila* may contain undesirable impurities and possesses properties that make it unsuitable for direct medicinal use. The primary objectives of *Shodhana* are to remove or reduce impurities, enhance the quality of the drug, improve its pharmaceutical acceptability, and make it safer for therapeutic use. Different purification media and procedures are prescribed in Ayurvedic classics, which are believed to modify the physicochemical properties of the mineral without adversely affecting its desired medicinal characteristics. From the Ayurvedic perspective, *Shodhana* reduces the harmful effects (*Doshas*) associated with the raw mineral and enhances its therapeutic utility.

In the present era, scientific validation of this traditional process is essential for ensuring the quality, safety, and standardization of Ayurvedic mineral preparations. Modern analytical techniques such as X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) provide valuable information regarding elemental composition and crystalline characteristics, thereby helping to assess the effect of *Shodhana* on *Manahsila* and supporting the scientific basis of the traditional purification process.

## MATERIAL AND METHODS

### *Manahsila Shodhana*

Reference-*Rasa Tarangini* 11/111

Principle- *Mardana*

Equipments required- *Khalva Yantra*, Spoon, Measuring jar, Weighing balance

### Ingredients

*Ashuddha Manahsila*: 100 gm

*Nimbu swarasa*: Q.S.

### Pre-Procedure

- The lemons were washed thoroughly with water and cleaned on the outside.
- Using a clean knife, each lemon was cut in half into equal pieces.
- Then, the lemon halves were placed in a lemon squeezer, and the juice was extracted.
- The juice was strained through a clean filter and the filtered liquid was collected as lemon juice.

### Pre-procedure

|                         |        |
|-------------------------|--------|
| Weight of <i>Nimbu</i>  | 500 gm |
| <i>Swarasa</i> obtained | 100 ml |

### Procedure

- Physical impurities such as stones and sand were removed manually.
- Ashuddha Manahsila* was placed in a clean *Khalva Yantra* and broken into small pieces.
- Sufficient quantity of lemon juice was added until the churna was completely immersed in the *swarasa*.
- Bhavana* (trituration) was carried out gradually for at least 6 hours each day.
- After the *swarasa* dried, fresh *swarasa* was added the next day and *bhavana* was continued.
- Bhavana* was repeated daily for a period of 7 days.
- After the final *bhavana*, it was washed 3-4 times with normal water.
- After washing, it was left to dry and once completely dry, *Shuddha Manahsila* was collected.

### OBSERVATION

- The color of *Ashuddha Manahsila* was reddish - brown with a slight blackish tinge, had a smooth shiny crystalline texture, and emitted a distinctive odor.

- It had the aroma of the *bhavana dravya* combined with the characteristic smell of *Manahsila*.
- The shiny particles were completely lost.
- After proper drying, yellowish - orange small flakes of *Shuddha Manahsila* were collected.

#### Precautions

- *Ashuddha Manahsila* was broken into small pieces before being subjected to the process.
- Trituration was performed carefully.
- The supernatant water used in *Manahsila Shodhana* was carefully separated.

#### RESULTS

To evaluate the effect of *Shodhana*, advanced instrumental analyses, including X-ray Fluorescence

(XRF) and X-ray Diffraction (XRD), were carried out on *Manahsila* samples before (*Ashuddha Manahsila*) and after *Shodhana* (*Shuddha Manahsila*) at the Department of Metallurgical and Materials Engineering, Indian Institute of Technology (IIT) Roorkee, Uttarakhand, India, using calibrated instruments and standard analytical protocols.

#### I. Results of *Manahsila Shodhana*

The initial weight of *Ashuddha Manahsila* was 100 g. After completion of the *Shodhana* process, the weight of the obtained *Shuddha Manahsila* was 92 g, indicating a total weight loss of 8 g during purification. This reduction in weight may be attributed to the removal of adhering impurities and the loss of extraneous materials during the *Shodhana* procedure.

#### II. Value of element in XRF of *Ashuddha Manahsila*

Table 1: Value of element in XRF of *Ashuddha Manahsila*.

| Component | Unit  | Result  |
|-----------|-------|---------|
| Al        | Mass% | 1.9955  |
| Si        | Mass% | 2.1262  |
| P         | Mass% | 0.0525  |
| S         | Mass% | 28.9289 |
| Cl        | Mass% | 0.0367  |
| K         | Mass% | 0.1251  |
| Ca        | Mass% | 1.5551  |
| Fe        | Mass% | 0.3932  |
| As        | Mass% | 64.4020 |
| Sr        | Mass% | 0.0345  |
| Sb        | Mass% | 0.3504  |

XRF elemental scanning of *Ashuddha Manahsila* shows As 64.40 mass% and S 28.92 mass% is seen in max.

quantity and Ca, Si, Sb etc. in descending quantity. (In Table no.1).

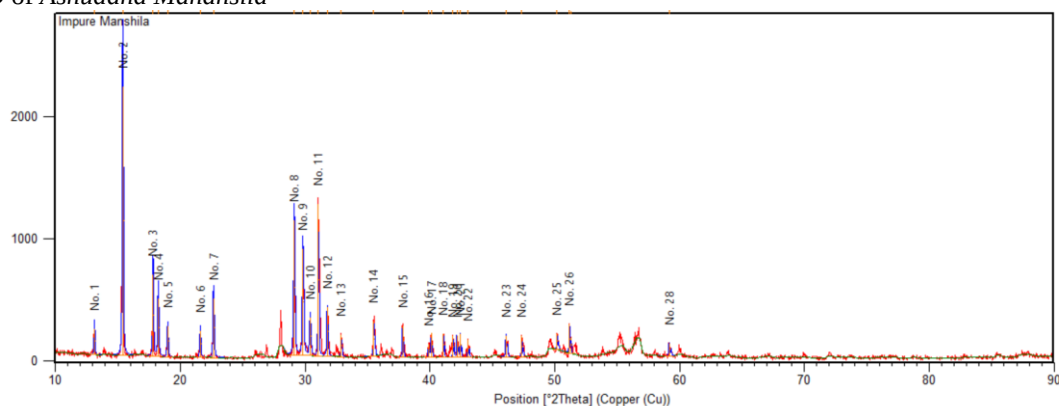
#### III. Value of element in XRF of *Shuddha Manahsila*

Table 2: Value of element in XRF of *Shuddha Manahsila*.

| Component | Unit  | Result  |
|-----------|-------|---------|
| Al        | Mass% | 0.0830  |
| Si        | Mass% | 0.2173  |
| S         | Mass% | 29.9604 |
| Cl        | Mass% | 0.0448  |
| K         | Mass% | 0.0980  |
| Ca        | Mass% | 0.0404  |
| Fe        | Mass% | 0.0256  |
| Zn        | Mass% | 0.0153  |
| As        | Mass% | 69.5152 |

XRF elemental scanning of *Shuddha Manahsila* shows As 69.51 mass% and S 29.96 mass% is seen in max.

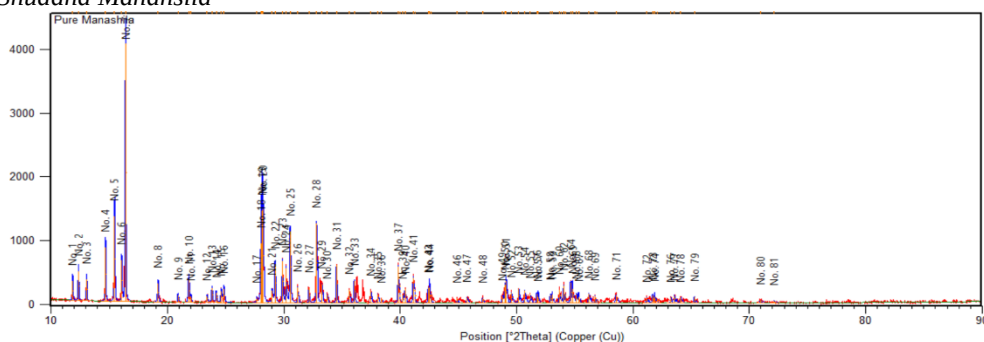
quantity and Ca, Si, Sb etc. in descending quantity. (In Table no. 2)

IV. XRD of *Ashuddha Manahsila*Fig. No. 1 XRD of *Ashuddha Manahsila*.Table 3: Showing XRD of *Ashuddha Manahsila*.

| Standard   |         |           | Impure <i>Manahsila</i> |         |           |
|------------|---------|-----------|-------------------------|---------|-----------|
| 2 $\theta$ | d-space | Intensity | 2 $\theta$              | d-space | Intensity |
| 13.13      | 6.73    | 10        | 13.12                   | 6.74    | 11        |
| 15.38      | 5.75    | 100       | 15.38                   | 5.75    | 100       |
| 17.82      | 4.97    | 33        | 17.81                   | 4.97    | 30        |
| 18.23      | 4.86    | 24        | 18.22                   | 4.86    | 21        |
| 18.99      | 4.66    | 13        | 18.98                   | 4.67    | 11        |
| 21.60      | 4.11    | 15        | 21.59                   | 4.11    | 10        |

The XRD analysis of *Ashuddha Manahsila* showed a close agreement with the standard reference (Card No. 999012101) (In fig. no.1 and Table no. 3). The major diffraction peaks may exhibit nearly similar 2 $\theta$  values, d-

spacing and relative intensities, suggesting that the primary crystalline phase may have remained unchanged. Minor variations in peak positions and intensities may be attributed to impurities or instrumental factors.

V. XRD of *Shuddha Manahsila*Fig. No. 2 XRD of *Shuddha Manahsila*.Table 4: Showing XRD of *Shuddha Manahsila*.

| Standard   |         |           | Pure <i>Manahsila</i> |         |           |
|------------|---------|-----------|-----------------------|---------|-----------|
| 2 $\theta$ | d-space | Intensity | 2 $\theta$            | d-space | Intensity |
| 13.13      | 6.73    | 10%       | 13.11                 | 6.69    | 8%        |
| 15.38      | 5.75    | 100%      | 15.46                 | 5.72    | 100%      |
| 17.82      | 4.97    | 33%       | 16.10                 | 5.5     | 30%       |
| 18.23      | 4.86    | 24%       | 16.28                 | 5.43    | 24%       |
| 18.99      | 4.66    | 13%       | 19.14                 | 4.63    | 12.5%     |
| 21.60      | 4.11    | 15%       | 21.78                 | 4.07    | 14%       |

The comparison of the major XRD peaks of the standard and *Shuddha Manahsila* showed a close agreement in 2 $\theta$  values, d-spacing, and relative intensities with the standard reference (Card No. 999010054) (In fig. no. 2

and Table no. 4). The good correspondence of the characteristic diffraction peaks may suggest that the crystalline phase of *Shuddha Manahsila* remained consistent with the reference standard. Minor variations

in peak positions and d-spacing may be attributed to instrumental error, slight lattice distortion, or subtle structural changes that may have occurred during the purification process. Overall, the XRD results may confirm the identity and phase purity of *Shuddha Manahsila*.

## DISCUSSION

*Manahsila* possesses *Tikta* and *Katu rasa*, which may contribute to its detoxifying and antimicrobial effects. Its *Laghu guna* make it suitable for topical application. The *Ushna veerya* of *Manahsila* may enhance anti-inflammatory activity. *Manahsila Shodhana* was performed using *Nimbu Swarasa* in accordance with traditional *Ayurvedic* procedures. The acidic character of *Nimbu Swarasa* may have aided in the elimination of contaminants and assisted to the detoxification of *Manahsila*, improving its purity and medicinal potential. In study, an 8% weight loss was observed after *Manahsila Shodhana*. This reduction may be due to removal of impurities along with minor handling and processing losses during washing, drying and transfer of the material. XRF analysis of *Manahsila* before and after *Shodhana* revealed an increase in the percentage of Arsenic and Sulphur, which are the principal constituents of *Manahsila*. Additionally, the levels of associated elements such as Al, Si, Ca, Fe, Sr, Sb were markedly reduced or became negligible after purification. The observed increase in As and S content may result from the removal of associated mineral impurities during *Shodhana* process, leading to a relative enrichment of the characteristic constituents. These findings may indicate improved purity and pharmaceutical suitability of *Manahsila*. XRD analysis was performed to assess the effect of *Shodhana* on the crystalline structure of *Manahsila*. Comparison of the diffraction patterns before and after *Shodhana* showed that the major characteristic peaks,  $2\theta$  values, and d-spacing remained largely consistent, indicating that the fundamental crystalline phase was preserved during purification. Minor variations in peak positions and intensities may be attributed to the removal of impurities, slight lattice distortion, or instrumental factors. Overall, the findings suggest that *Shodhana* may remove undesirable impurities without causing significant changes in the crystalline structure of *Manahsila*, thereby supporting the traditional *Ayurvedic* purification process.

## CONCLUSION

The present study demonstrates that the purification (*Shodhana*) process of *Manahsila* can effectively produce *Shuddha Manahsila* while retaining its characteristic crystalline structure. Physicochemical and instrumental analyses indicate that the purified sample exhibits properties consistent with the reference standard. The XRD results may confirm the identity and phase purity of *Shuddha Manahsila*, while other analytical findings suggest that the purification process may reduce impurities without causing any significant alteration in its fundamental structure. These observations support the

importance of the traditional *Shodhana* process in improving the quality and standardization of *Manahsila*. However, further advanced analytical and pharmacological studies may provide additional evidence regarding its safety, efficacy, and therapeutic potential.

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