ABSTRACT

Mineral drugs play an important role in Ayurvedic therapeutics. Makshika, Copper pyrite is considered as Maharasa in Rasasastra. It is described as one among best Rasayananas in classical literatures. This study is an attempt to standardize the process of preparation of Swarna Makshika bhasma. The process commences with selection of raw material, which is the prime and crucial step ahead. Ayurveda Pharmacopoeia of India standards and classical methods were used for the same. Swarna Makshika bhasma was prepared as per the reference from Ayurveda Prakasha (Shodhana) and Rasarata Samucchayam (Marana). Shodhana (purification) was done by open heating in an iron pan by adding Saindhava (rock salt) and lemon juice. Five Varaha puta with 600°C quantum of heat was opted for Marana. The process was performed using an Electric Muffle furnace. During each step classical methods for analyzing the product (Bhasma) was done which ensured the proper Bhasmeekarana. Swarna Makshika bhasma was then subjected to X-ray Diffraction study which reveals the presence of both haematite (58%) and magnetite (40%) in it and the structural representation of them in Bhasma was also noted. XRF study conducted found that the Bhasma possesses 75.54% Iron and rest of the elements within its permissible limits which ultimately unwrap its safety. Scanning Electron Microscope study gave the particle size of Bhasma to be 2microns (50000 X magnification). The particle size recorded can be considered as the desired specification of the final Bhasma.

KEYWORDS: Swarna Makshika, Standardization, Therapeutics, XRD.

INTRODUCTION

The prime objective of pharmaceutical study is to produce safe, effective and quality drugs. In this era of globalization there lies a need and necessity to explore the scientific rationale and basics behind the therapeutics in Ayurveda. The scientific data thus generated will eventually lead to the acceptance of our drugs in global market. The pharmaceutical study in Ayurveda particularly Rasasastra commences with the identification of genuine raw materials, selection of suitable processes which alters the physico-chemical and metallurgical properties. This will eventually bestow the drug with characteristic features like low therapeutic doses, safety, longer shelf life. Makshika (Copper pyrite) is one of the material used since long, having therapeutic importance. From the Samhita period to the modern era of science and technology Makshika (Copper pyrite) has occupied an important position in Ayurvedic system. It is a mineral of Copper, Iron and Sulphur. Therapeutic description of Makshika is found in the literatures of Samhita period but its detailed pharmaceutical description is found in Rasasastra literatures. A standardized method in the preparation and its physico chemical analysis of Swarna makshika bhasma will be of utmost important in the current scenario.

MATERIALS AND METHODS

Swarna Makshika

The raw Swarna makshika was obtained from a genuine source at Palakkad. Then its consanguinity was tested via classical as well as Ayurveda Pharmacopoeia of India[1] parameters. In classics it was described that a sample with qualities[2] guru (heavy), Snigdha (sliminess), black in colour with golden tints, on rubbing black in colour was termed as a genuine sample. The below said physical characters are also helped in identifying the genuine sample.

Physical characters

- Colour - Blackish mineral with golden lustre
- Odour - Nil
- Taste - Tasteless
- Lustre - Metallic
- Streak - Dark green
- Hardness - 3.5 - 4 (Mohs scale)
- Streak - Black
As per API parameters for a sample of *Swarna makshika* to be acceptable for therapeutic purpose the percentage of copper should be above 5%. An Atomic Absorption Spectroscopy was done which revealed that the selected sample had a copper content 8.1%. So the sample was selected for *Bhasma* preparation.

*Saindhava Rock salt*, *Gandhaka* (Sulphur) were obtained from a reputed Ayurveda raw drug shop at Thiruvananthapuram. Lemon and *Matulunga* (*Citrus medica*) are obtained from Kollam.

**Pharmaceutical Processing**

*Swarna makshika bhasma nirmanam* (incineration) involves two steps:

a) *Shodhana* (Purification)
b) *Maranam* (incineration)

**Shodhana of Swarna makshikam**

*Shodhana* was done as per the reference from Ayurveda Prakasa.[3] Open heating of powdered *Swarna makshika* with one third of its quantity of rock salt along with lemon juice sprinkled at regular intervals. The process was continued till the iron pan get red hot (*Tamra varnam ayo*) and the drug turned a coppery shade. Then it was allowed to cool, washed 3-4 times thoroughly in running water to make it devoid of *Lavana rasa* (salty taste). Then dried well and used for next step.

**Table 1: Shodhana of Swarna makshika**

<table>
<thead>
<tr>
<th>Drug</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Swarna makshika</td>
<td>300 g</td>
</tr>
<tr>
<td>Saindava (rock salt)</td>
<td>100 g</td>
</tr>
<tr>
<td>Lemon juice</td>
<td>100 ml</td>
</tr>
</tbody>
</table>

**Procedure**

300 g of powdered raw *Swarna makshika* and 100 g of powdered *Saindhava* were mixed well and taken in an iron vessel and placed over coal fire. Strained Lemon juice was added to it little by little thereafter and stirred continuously with an iron spatula. The process was continued till the iron pan get red hot and the whole mass turned to coppery colour. Then the contents were allowed to cool and was washed to remove the salt content.

**Raw materials for Swarna makshika shodhana**

![Saindhava](image1.png) ![Lemon](image2.png) ![Swarna Makshika](image3.png)

**Shodana of Swarna makshika**

<table>
<thead>
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<tr>
<td>Lemon juice</td>
<td>100 ml</td>
</tr>
</tbody>
</table>

**Result**

Initial Weight: 300g  
Final Weight: 285g (after washing)  
Loss of Drug: 15g  

Necessary precautions like wearing mask, gloves etc., should be taken during the process of purification as heating generates sulphur fumes in large quantity that may cause suffocation.

**Maranam of Swarna makshika**

The *Marana* was done as per the reference from Rasaratna Samuchhayam.[4] For the process of Marana, equal amount of purified *Swarnamakshika* and *Gandhaka* were taken and triturated well with *Matulunga rasa* till a homogenous paste was formed. The quantity of *Gandhaka* in the present study was taken in reference with Rasamrutham.[5] Accordingly the quantity of *Gandhaka* can be reduced for subsequent *Putas* which gives the characteristic colour to the *Bhasma*.

After triturating, small pellets of uniform size and thickness were prepared and dried well. Pellets were kept inside a *Sharava* (shallow earthen disc)
and another Sharava was inverted over it. The joint between the two disc was sealed with a cloth smeared with Fuller’s earth seven times and dried in sunlight.

The properly dried and sealed Sharava was subjected to Puta by placing in Muffle furnace. The temperature set was 600°C (Varah puta) and maintained for one hour. The process was repeated Bhavana of Shodhitha swarna makshika upto palatalisation for five times, till Bhasma of required standards were obtained.

The needful for Bhasma preparation were
1. Shoditha swarna makshikam
2. Shoditha Gandhakam (Obtained as Gandhaka mani- kurma puta method)[5]
3. Matulunga rasam (Citrus medica)

Details of Puta

Table 2: Marana of Swarna makshika

<table>
<thead>
<tr>
<th>No. of Putas</th>
<th>Quantity of Marana dravya</th>
<th>Quantity of Gandhaka</th>
<th>Temperature</th>
<th>Time for attaining temperature</th>
<th>Initial weight of Chakrikas</th>
<th>Final weight after Maranam</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150</td>
<td>150 (equal)</td>
<td>600°C</td>
<td>1 hours</td>
<td>278 gms</td>
<td>139.8 gms</td>
</tr>
<tr>
<td>2</td>
<td>139.8</td>
<td>75 (half)</td>
<td>600°C</td>
<td>1.03 hours</td>
<td>211.6 gms</td>
<td>125.750 gms</td>
</tr>
<tr>
<td>3</td>
<td>125.750</td>
<td>37.5 (one fourth)</td>
<td>600°C</td>
<td>1 hours</td>
<td>178.470 gms</td>
<td>114.550 gms</td>
</tr>
<tr>
<td>4</td>
<td>114.550</td>
<td>Not added</td>
<td>600°C</td>
<td>1.15 hours</td>
<td>149.96 gms</td>
<td>101.520 gms</td>
</tr>
<tr>
<td>5</td>
<td>101.520</td>
<td>Not added</td>
<td>600°C</td>
<td>1.2 hours</td>
<td>121.95 gms</td>
<td>86.75 gms</td>
</tr>
</tbody>
</table>

Initially the Gandhaka was added in same quantity as that of Makshika. But on later Putas the proportion of Gandhaka was reduced to half and one forth. The last two Puta was done without Gandhaka.

Results
Initial quantity of Swarna makshika: 150 g
Final quantity of Swarna makshika bhasma: 86.75 g

The Swarna makshika, showed a loss in weight from raw to Maarittha loha bhasma. The colour had changed to dark brick red and there occurred appreciable change in the particle size.

ANALYTICAL STUDY
The Swarna makshika bhasma prepared was subjected to classical method of analysis- Bhasma pareeksha For Makshika bhasma an extra Bhasma pareeksha is told, which is Avaamitwa i.e., when the Bhasma was placed on the tongue, it should not produce any nausea or vomiting sensation.
Table 3: Classical evaluation of Swarna makshika Bhasma

<table>
<thead>
<tr>
<th>Puta</th>
<th>Nischandratwam</th>
<th>Rekha purnada</th>
<th>Varitaratwam</th>
<th>Nisvathutwam</th>
<th>Avami</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Absent</td>
<td>80%</td>
<td>15%</td>
<td>Metallic taste</td>
<td>Not attained</td>
</tr>
<tr>
<td>2</td>
<td>Absent</td>
<td>Present</td>
<td>25%</td>
<td>Metallic</td>
<td>Not attained</td>
</tr>
<tr>
<td>3</td>
<td>Present</td>
<td>Present</td>
<td>40%</td>
<td>Metallic taste decreased</td>
<td>Not attained</td>
</tr>
<tr>
<td>4</td>
<td>Present</td>
<td>Present</td>
<td>55%</td>
<td>Taste further diminished</td>
<td>Present</td>
</tr>
<tr>
<td>5</td>
<td>Present</td>
<td>Present</td>
<td>75%</td>
<td>No metallic taste</td>
<td>Present</td>
</tr>
</tbody>
</table>

From the table it was clear that Nischandratwam was present for the Bhasma from the third Puta. Rekhapurnada was developed after second Puta only. Varitaratwam was seen after fourth Puta. The Bhasma had satisfied the Avaami pareeksha from the fourth Puta.

Classical evaluation of Bhasma

a) Rekhapurnada

b) Vaaritaratwa

Swarna makshika - XRD

XRD or X-ray diffraction is a rapid analytical technique primarily used for the phase identification of a crystalline material and can provide information on the unit cell dimensions. The present study was conducted at Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, Tamil Nadu.

X-ray powder diffraction pattern of Makshika bhasma was done in INEL make XRG 3000 diffractometer with monochromatic Cu Ka1 radiation (\(\lambda =1.54056\) Å) equipped with curved position detector (model CPS590). The X-ray data collected in the range of 10 to 100 degrees of two theta with step size of 0.012 degrees.

Results and Discussions

The powder pattern shows sharp peaks indicated that the powder was of crystalline nature. The peaks were identified and indexed for Hematite and Magnetite using ICDD JCPDF cards # 033-0664 and # 01-080-6402. To quantify the phases present in the powder, the powder data was analysed using Rietveld methodology. The final Rietveld plot of the Swarna makshika bhasma powder was shown in graph.2.

The volume fraction of the chalcopyrite was less than 2%, whereas 58% Hematite and 40% of Magnetite phases present in these powder. The calculated lattice parameter almost equal to the previous Hematite and Magnetite phases with small difference was observed.

The refined lattice parameter of chalcopyrite phase is found to be \(a = 5.13947(8), c = 10.6734(4)\) with the tetragonal crystal structure (space group: \(I-4 \overline{2} d\)). The lattice parameter of rhombohedral Hematite phases was found to be \(a = 5.03832, c = 13.784105\) the refined lattice parameter of face centered cubic Magnetite phase was found to be \(a = 8.35435\) Å.

Powder s shows three phase mixture of chalcopyrite, Hematite and Magnetite phases
The crystal structure of chalcopyrite phase was shown below

Crystal structure of chalcopyrite in (a) ball and stick and (b) polyhedral model. Blue color sphere c) represents Cu and golden yellow is Fe and yellow colour sphere represents sulphur atoms.

**X Ray Fluorescence Analysis**

XRF is a non-destructive analytical technique used to determine the elemental composition of materials like rocks, minerals, sediments and fluids. It works on the wavelength-dispersive spectroscopic principles that are similar to electron microprobe (EPMA) The relative ease and low cost of sample preparation, and the stability and the ease and use of x-ray spectrometers make this one of the most widely used method of analysis of major and trace elements in rock, minerals and sediments. The XRF method depends on fundamental principle include interaction between electron beams and x rays with samples.

XRF analysis done at X-ray Fluorescence Lab at National Centre For Earth Science Studies, Trivandrum reveals the following data.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Swarna makshika bhasma</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>9.83</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.18</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>2.98</td>
</tr>
<tr>
<td>MnO</td>
<td>ND</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>75.54</td>
</tr>
<tr>
<td>CaO</td>
<td>1.29</td>
</tr>
<tr>
<td>MgO</td>
<td>0.21</td>
</tr>
<tr>
<td>Na₂O</td>
<td>1.02</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.90</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.17</td>
</tr>
<tr>
<td>SO₃</td>
<td>3.19</td>
</tr>
<tr>
<td>BaO</td>
<td>0.80</td>
</tr>
<tr>
<td>Total</td>
<td>96.11</td>
</tr>
</tbody>
</table>
SEM ANALYSIS

Scanning Electron Microscopy was done at Sree Chitira Thirunal Institute of Science and Technology, Mudavanmugal, Thiruvananthapuram. SEM is a valuable technique for characterizing the surface morphology of all kinds of samples and materials. In this study it was revealed that the particle size of the raw material was between 6-8µ while that for the Bhasma particles was 2µ (50,000X magnification)

SEM images of Swarna makshika in 10,000 and 50,000 X magnification

OBSERVATION AND ANALYSIS

The very first step in the standardisation was to collect genuine raw materials. Both classical and modern analytical parameters of standardisation had done to procure the genuine samples. Makshika was obtained from a genuine source and both the classical and modern analytical parameters were used to unveil the genuine sample.

Moving to the processing section the raw Makshika was found to be hard and difficult to get powdered. Later after purification the main change observed was colour of Makshika had changed to blackish coppery and reduction in hardness and particle size. On heating the mixture offensive sulphurous had occurred which was highly suffocating. The process was continued till the whole mass attained coppery colour. The relative weight loss was 5%.

For the Maarana of Swarna makshika, Gandhaka was used as the Maaraka dravya and Bhavana was done with Matulunga swarasm (Citrus medica). A total of 5 Puta was done to obtain the Bhasma of brick red colour. In the present study reduction of Gandhaka in subsequent Puta was adopted which seem to be effective. The raw Makshika was hard in consistency, but after Shodhana it became soft. The temperature selected was 600°C and classical Bhasma pareeksha was used for determining well formed Bhasma. The % of weight loss during Swarna makshika marana was 42.166%.

Heavy metal analysis of the formulation was carried out by Atomic Absorption Spectroscopy at Drug Standardisation Unit, Govt.Ayurveda College Thiruvananthapuram. Heavy metals like Cadmium, Lead, Zinc analysis were carried out and all of them were found within the permissible limit.

Instrumental analytical methods like XRD, XRF, SEM of the Bhasma done which ultimately reveal the chemical nature, composition and fineness of the preparation. The scanning electron microscope (SEM) analysis done at Sree Chitira Thirunal Institute of Science and Technology, TVPM concluded that the process of Shodhana and Maranam imparted characteristic change in the particle size of mineral drug – Makshika. The particles are found as clusters under the magnification 50,000X with 2µ size. There is a significant reduction in the size which allows the phenomenon of Rekhapurna and Varitararwa to develop. Reduction in particle size facilitates absorption and assimilation of the Bhasma in the system. The particle size recorded can be characterized as the desired specification of the final Bhasma. The XRD was studied at Indira Gandhi Centre For Atomic Research (IGCAR), Kalpakkam, Tamil Nadu. The chalcopyrite composition was less than 2% and that of haematite 58%, and magnetite 40%. The analysis reveals the chemical changes happening to mineral drugs during the process of Bhaavanas. Makshika was converted to a more stable and acceptable form Haematite.

The XRF analysis was conducted at National Centre for Earth Studies (NCESS), Akkulam. XRF analysis reveals the elemental composition of the mineral drugs. The iron portion was prominent in the samples of Makshika (75.54 and 70.70 for Swarna makshika).

Available online at: http://ijapr.in
Classical Method of Evaluation of Bhasma

The final Bhasma was analyses for the quality control as described in Ayurvedic texts as follows and found suitable.

1. Nischandratva: The Bhasma will take in a Petri dish and observed for lustre in day light through magnifying glass. No lustre will be observed in a properly formed Bhasma.

2. Rekhapurnatvat: A pinch of Bhasma is taken between the thumb and index finger and rubbed. The Bhasma will enter into the lines of fingers and is not easily washed out from the cleavage of the lines.

3. Varitaratva: A small amount of the prepared Bhasma will be sprinkled over the still water in a beaker. It is found that the Bhasma particle will float over the surface of water.

4. Nisvadutvam: The prepared Bhasma was found to be tasteless when a small amount was kept on the tongue.

5. Amla pariksha (For Tamra and Swarna makshika): A pinch of prepared Bhasma was mixed with a little amount of Dadhi (curd) in a clean and dry Petri dish and observed for any colour change. No colour change of Dadhi was observed. The same procedure will be followed with lemon juice taken in test tube and the same result will be observed.

6. Avami (For Tamra and Swarna makshika): Ingestion of 5-10 mg of the Bhasma did not produce any nausea / vomiting.

CONCLUSION

The Rasasatra therapeutics has to be revalidated with the existing techniques which help us to affirm the rationale behind the each and every process adopted by our Acharyas. This will eventually make our science much more acceptable to this world as well as to the coming generation. The concept behind Shodhana and Marana can be well revealed in this study by classical as well as analytical methods XRD, XRF and SEM analysis. The XRD structure representation of a Bhasma seems to be first of its kind and should be considered for further studies.

ACKNOWLEDGEMENT

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*Address for correspondence
Dr Raji.R.Nair
PG Scholar,
Department of Rasasastra and Bhaishajya Kalpana,
Thiruvananthapuram, Kerala, India.
Mobile: 9447791629
Email: rajiranil2009@gmail.com

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