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Research Article

PREPARATION AND PHYSICOCHEMICAL CHARACTERISATION OF *SWARNA MAKSHIKA BHASMA*

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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 *Rasayanas* 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 Rasaratna 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 600^0C 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* posses 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.

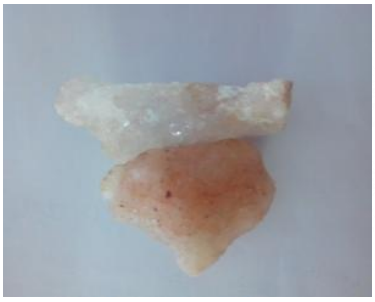
Table 1: *Shodhana* of *Swarna makshika*

Drug	Quantity
Swarna makshika	300 g
Saindava (rock salt)	100 g
Lemon juice	100 ml

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*

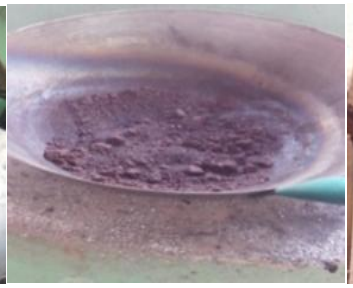


Saindhava

Lemon

Swarna Makshika

Shodana* of *Swarna makshika



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^{0}c (*Varah puta*) and maintained for one hour. The process was repeated 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*)

Bhavana of _Shodhitha swarna makshika_ upto palatalisation



Details of _Puta_




Table 2: _Marana_ of _Swarna makshika_

No. of *Putas*	Quantity of *Marana dravya*	Quantity of *Gandhaka*	Temperature	Time for attaining temperature	Initial weight of *Chakrikas*	Final weight after *Maranam*
1	150	150 (equal)	600℃	1 hours	278 gms	139.8 gms
2	139.8	75 (half)	600℃	1.03 hours	211.6 gms	125.750 gms
3	125.750	37.5 (one fourth)	600℃	1 hours	178.470 gms	114.550 gms
4	114.550	Not added	600℃	1.15 hours	149.96 gms	101.520 gms
5	101.520	Not added	600℃	1.2 hours	121.95 gms	86.75 gms

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 *Maaritha loha bhasma*. The colour had changed to dark brick red colour 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*

Puta	***Nischandratwam***	***Rekha purnada***	***Varitar atwam***	***Nisvathutwam***	***Avami***
1	Absent	80 %	15%	Metallic taste	Not attained
2	Absent	Present	25%	Metallic	Not attained
3	Present	Present	40%	Metallic taste decreased	Not attained
4	Present	Present	55%	Taste further diminished	Present
5	Present	Present	75%	No metallic taste	Present

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.

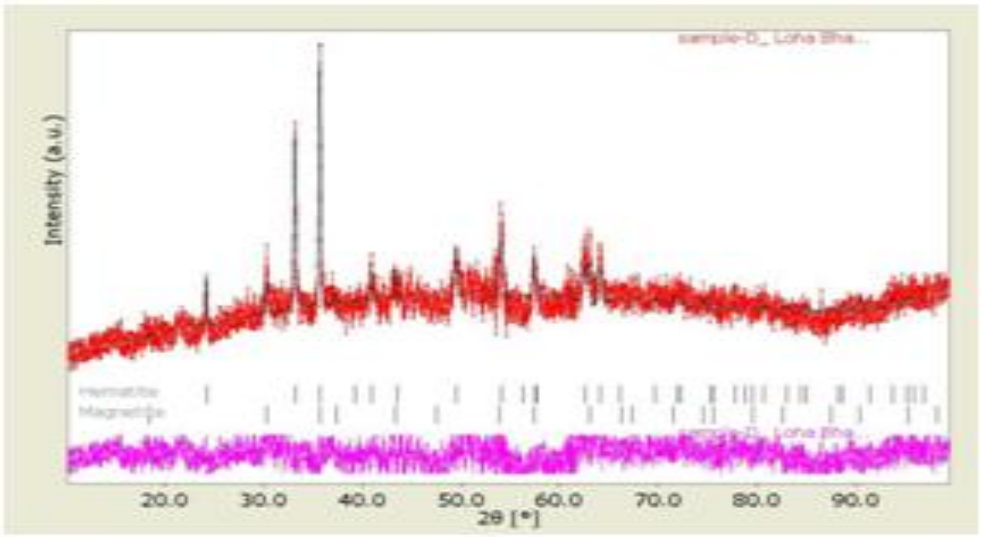
X-ray powder diffraction pattern of *Makshika bhasma* was done in INEL make XRG 3000 diffractometer with monochromatic Cu Ka1 radiation (λ =1.54056 Å) equipped with curved position detector (model CPS590). The X-ray data collected in the range of 10 to 100 degrees of two theta with step size of 0.012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

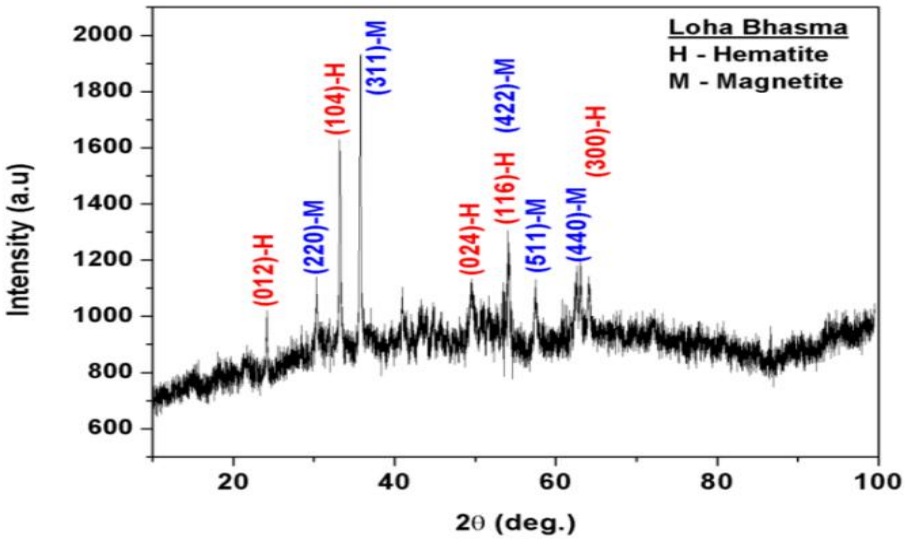
The volume fraction of the chalcopyrite was less than 2%, whereas 58% Hematite and 40% of Magnetite phases present in these powder. The calculated lattice parameter almost equal to the previous Hematite and Magnetite phases with small difference was observed.

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 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.

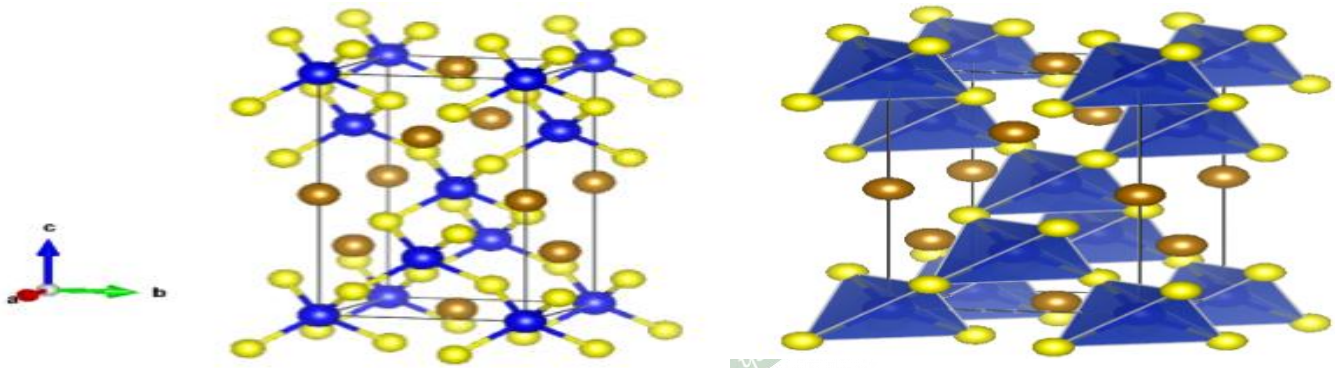


Powder s shows three phase mixture of chalcopyrite, Hematite and Magnetite phases

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 Flouorescence Lab at National Centre For Earth Science Studies, Trivandrum reveals the following data.

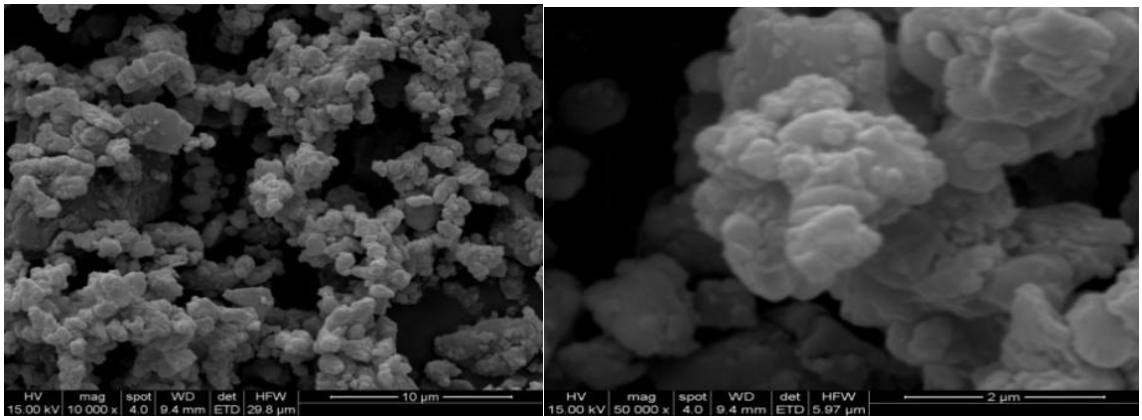
Sample	***Swarna makshika bhasma***
SiO_2	9.83
TiO_2	0.18
Al_2O_3	2.98
MnO	ND
Fe_2O_3	75.54
CaO	1.29
MgO	0.21
Na_2O	1.02
K_2O	0.90
P_2O_5	0.17
SO_3	3.19
BaO	0.80
Total	96.11

SEM ANALYSIS

Scanning Electron Microscopy was done at Sree Chitira Thirunal Institute of Science and Technology, Mudavanmugal, Thiruvananthapura. 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 suphurous 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 600^0C 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 hap penning 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*.

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. ***Rekhapurnatvam:*** 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. ***Varitaratvam:*** 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 re validated 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.

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