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Short Communication

X-Ray Diffraction of different samples of *Swarna Makshika Bhasma*

Ramesh Kumar Gupta, Vijay Lakshmi[1], Chandra Bhushan Jha[2]

Department of Rasa Shastra and Bhaishajya Kalpana, [1]Department of Prasooti Tantra, Government Ayurvedic College, Varanasi, [2]Faculty of Ayurveda, Institute of Medical Sciences, Banaras Hindu University, Varanasi, Uttar Pradesh, India

Abstract

Introduction: *Shodhana* and *Marana* are a series of complex procedures that identify the undesirable effects of heavy metals/minerals and convert them into absorbable and assimilable forms. Study on the analytical levels is essential to evaluate the structural and chemical changes that take place during and after following such procedures as described in major classical texts to understand the mystery behind these processes. X-Ray Diffraction (XRD) helps to identify and characterize minerals/metals and fix up the particular characteristics pattern of prepared *Bhasma*. **Aim:** To evaluate the chemical changes in *Swarna Makshika Bhasma* prepared by using different media and methods. **Materials and Methods:** In this study, raw *Swarna Makshika*, purified *Swarna Makshika* and four types of *Swarna Makshika Bhasma* prepared by using different media and methods were analyzed by XRD study. **Results:** XRD study of different samples revealed strongest peaks of iron oxide in *Bhasma*. Other phases of Cu_2O, FeS_2, Cu_2S, $FeSO_4$, etc., were also identified in many of the samples. **Conclusion:** XRD study revealed that *Swarna Makshika Bhasma* prepared by *Kupipakwa* method is better, convenient, and can save time.

Key words: *Bhasma*, *Makshika*, *Marana*, *Shodhana*, X-ray diffraction

Introduction

Ayurveda and other traditional medicines mainly depend on herbal, herbo-mineral formulations; have to change their track and method of approach to convince the scientific world. Some recent criticism from the West against the metallic preparations has created uproar from the Ayurvedic fraternity globally. An analytical study is one of the vital parts for drug standardization in traditional systems of medicine helps to interpret the pharmacokinetics and pharmacodynamics of Ayurvedic drugs.

Physico-chemical analysis provides objective parameters to fix up the standards for quality of raw drugs as well as finished products. Since *Rasa Shastra* has physics and chemistry as its close ally, there is scope to seek laws of chemistry and physics for providing a relationship for changes that take place in the pharmaceutical process.[1] In depth knowledge of imaging techniques and familiarity with the fundamental properties of matter are providing invaluable support for mapping the structure and function of drugs at all levels. Recent advances in data gathering techniques such as X-ray diffraction (XRD), field emission scanning electron microscopy, energy dispersive X-ray analysis provide an unprecedented view of the structure as well as cell function of the drug at the molecular and atomic level. These techniques are used in Ayurvedic pharmaceutical industries to characterize the raw material and final products and to establish this ancient science on modern scientific parameters. Hence, these tests can be put parallel to Ayurvedic *Bhasma Pariksha* (test) for ensuring genuine *Bhasma* production. Considering this, an effort has been made to analyze the raw *Swarna Makshika*, purified *Swarna Makshika* and four samples of *Swarna Makshika Bhasma* through XRD study. Prior to subjecting the material to XRD study, attempts were made to examine the *Bhasma* through classical parameters of analysis. In this study, emphasis has been given to find out the chemical changes takes place in *Swarna Makshika Bhasma* prepared by different methods by following X-Ray Diffraction.

Address for correspondence: Dr. Ramesh Kumar Gupta, Lecturer, Dept. of RS and BK, Government Ayurvedic College, Varanasi - 221 002, Uttar Pradesh, India.
E-mail: rameshguptabhu@gmail.com

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Materials and Methods

Materials and methods used in different samples of *Swarna Makshika Bhasma* preparations are based on availability, descriptions in *Rasa Shastra* classics, traditional values, and expert opinion. Raw *Makshika* is procured from the Ayurvedic Pharmacy of IMS, BHU. The raw drug is identified on the basis of its *Grahya Lakshanas* (acceptable characters) as mentioned in *Rasa* literatures,[2,3] and experts opinion.

Shodhana

Raw *Swarna Makshika* was taken in a clean and dry *Khalva Yantra* (mortar), pounded well to prepare fine powder, shifted to a clean and dry iron pan and subjected to intense heat at about a temperature of 750–900°C. The iron pan was then closed with an iron lid to avoid loss of material due to dusting. This process was continued for 3 days after complete cessation of sulfur fumes and until the mixture became red like fire.[4]

Marana

Four samples of *Swarna Makshika Bhasma* were prepared by following classical guidelines as described in Ayurveda classics.

Sample 1

Shodhita Swarna Makshika was triturated with lemon juice, and *Chakrika* (pellets) were made. Properly dried and weighed *Chakrikas* were arranged in a *Sharava*, closed by another *Sharava* and sealed by cloth smeared with clay. Properly sealed and dried *Sharava Samputa* was subjected to *Puta* system of heating. Twelve numbers of *Puta* were required to produce genuine *Bhasma*.[5]

Sample 2

Shodhita Swarna Makshika was mixed with equal amount of *Shodhita Gandhaka* and triturated with lemon juice; pellets were made and subjected to *Puta* system of heating. From second *Puta* onwards the amount of *Gandhaka* was taken half of the *Swarna Makshika*. Total 11 *Puta* were required to prepare *Swarna Makshika Bhasma*.[6]

Sample 3

Shodhita Swarna Makshika was mixed with 1/8th part of *Shodhita Hingula* and triturated with lemon juice; pellets were made and subjected to *Puta* system of heating. Total 09 *Puta* were required to prepare *Swarna Makshika Bhasma*.[7]

Sample 4

Shodhita Swarna Makshika was added with *Kajjali* and triturated with lemon juice till the material became homogenous and dried. The mixture was subjected for *Kupipaka* for 10 h. After breaking the *Kupi*, prepared *Swarna Makshika Bhasma* was collected from the bottom and *Rasa Sindura* was collected from the neck. Material collected from the bottom is further subjected to 6 *Putapaka* to prepare *Swarna Makshika Bhasma*.[8]

Analysis of samples

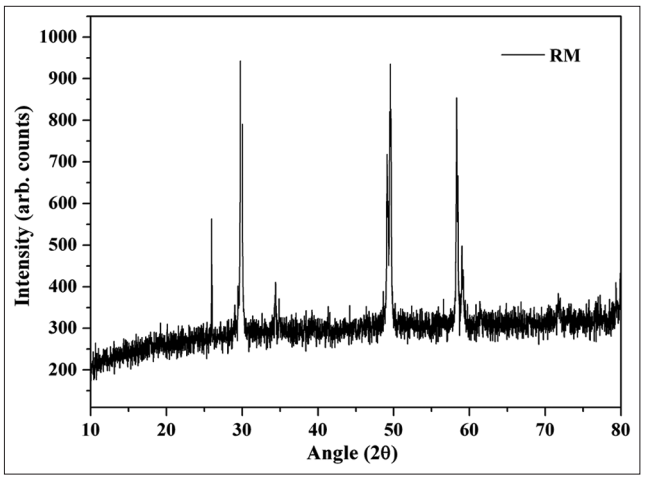
Samples of raw *Swarna Makshika*, *Shodhita Swarna Makshika*, and four samples of *Swarna Makshika Bhasma* were labeled and analyzed by XRD. The graph of each sample after comparing with Joint Committee on Power Diffraction Standards (JCPDS) data is illustrated in this study.

Observations and Results

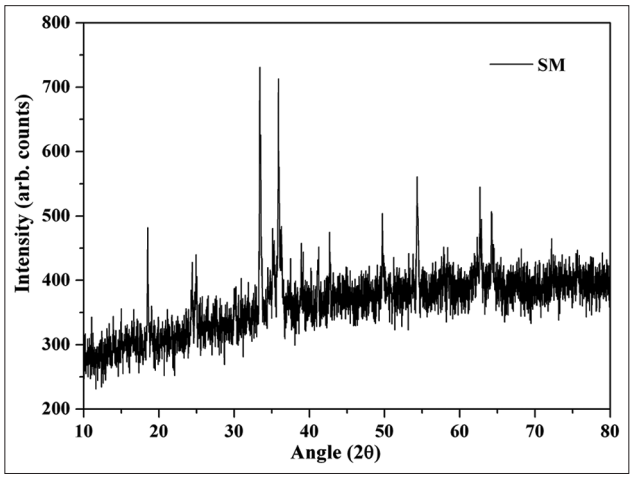
XRD study revealed that the strongest peaks identified in the raw material after comparing with JCPDS data was $CuFeS_2$ [Graph 1 and Table 1]. After *Shodhana*, the three highest peaks were identified as FeS_2, Fe_2O_3, and $FeSO_4$. Other peaks identified in *Shodhita Swarna Makshika* were Cu_2S and CuO [Graph 2 and Table 2]. Many complex compounds are also formed in *Shodhita Swarna Makshika*, but it is very difficult to detect them. Strongest peaks identified in sample 1 [Graph 3 and Table 3] sample 2 [Graph 4 and Table 4] sample 3 [Graph 5 and Table 5] of *Swarna Makshika Bhasma* is Fe_3O_4. In sample 4 of *Swarna Makshika Bhasma*, after *Kupipaka* [Graph 6 and Table 6] the peaks of $CuFeS_2$ again reappear and in the same sample, after *Putapaka* strong peaks of Fe_2O_3, Cu_2O, and $FeSO_4$ were identified [Graph 7 and Table 7].

Discussion

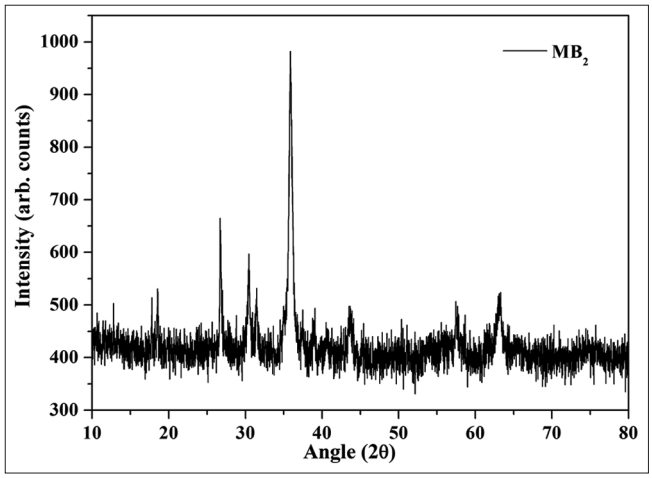
XRD of raw material reveals that the peaks obtained are corresponding to the peaks of $CuFeS_2$ in JCPDS file and hence the material is identified as copper pyrite. The strongest peak identified in *Shodhita* material (SM) was FeS_2. Other strong phases identified were Cu_2S, Fe_3O_4, $FeSO_4$, and SiO_2. The strongest peak identified in sample MB_1 was Fe_3O_4. In this sample, second and third strong peaks were identified as Fe_3O_4 and $FeSO_4$. The strongest peak identified in sample MB_2 was Fe_3O_4. Other strong peaks in MB_2 were identified as CuS and $FeSO_4$. Highest peak identified in MB_3 was Fe_3O_4. Other peaks identified in the sample were Cu_2O, $FeSO_4$, and Fe_2O_3. In partially prepared *Swarna Makshika Bhasma* (MBK_4) most of the highest peaks were identified as $CuFeS_2$. Regain of $CuFeS_2$ after *Kupipaka* was very surprising. As we know, SM contains mainly FeS_2, Cu_2S, Fe_3O_4, and $FeSO_4$. During *Kupipaka*, excess sulfur gets evaporated in the form of oxides of sulfur. Some of the sulfur reacted with mercury and converted into *Rasa Sindura* and some part of sulfur still remain un-reacted in the bottom of the bottle. On a specific temperature and conditions, this unreacted sulfur may react with copper and iron and get converted into copper pyrite. After further *Putapaka* of partially prepared *Swarna Makshika Bhasma*, highest



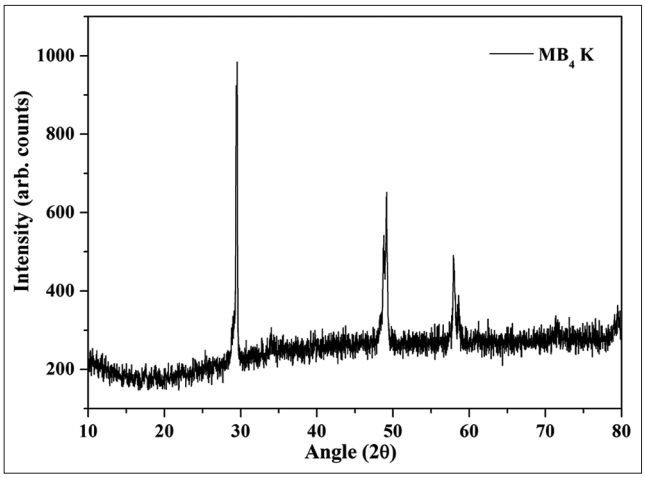
Graph 1: X-ray diffraction study of raw *Swarna Makshika*



Graph 2: X-ray diffraction study of Shodhita *Swarna Makshika*

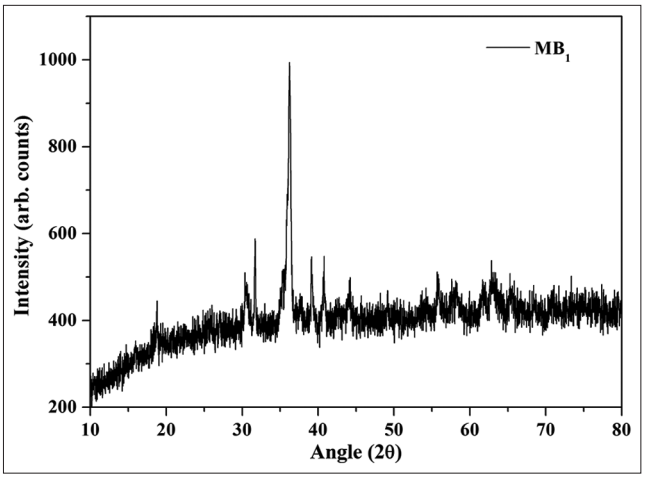


Graph 4: X-ray diffraction study of *Swarna Makshika Bhasma* (MB_2)

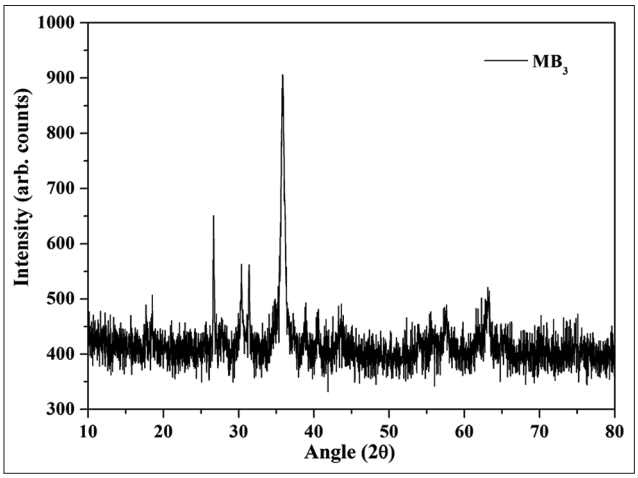


Graph 6: X-ray diffraction study of *Swarna Makshika Bhasma* (MB_4K)

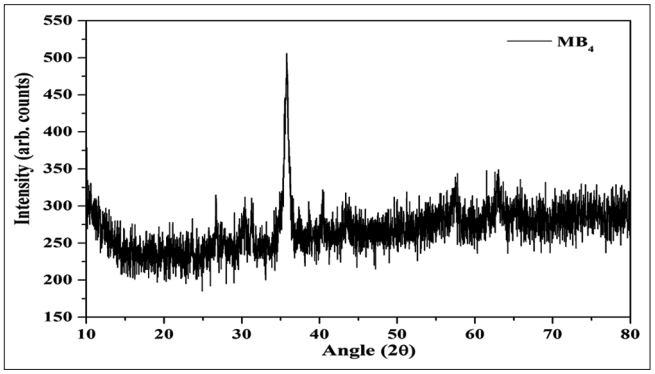
peaks identified were Fe_2O_3, Cu_2O, and $FeSO_4$. Hence, many small peaks are seen in all samples, but these small peaks are very difficult to identify. According to the diffraction principle, small



Graph 3: X-ray diffraction study of *Swarna Makshika Bhasma* (MB_1)



Graph 5: X-ray diffraction study of *Swarna Makshika Bhasma* (MB_3)



Graph 7: X-ray diffraction study of *Swarna Makshika Bhasma* (MB_4)

peaks are quantitatively very poor. On XRD report of the *Bhasma*, it can be assumed that the small peaks observed may be the trace elements or their compounds that may possibly incorporate into the prepared *Bhasma* due to repeated *Bhavana* (levigation) with herbal juices and firing in presence of oxygen and sulfur.

Table 1: The X-ray diffraction of raw *Swarna Makshika*

Compounds	Two theta	Intensity	Value of *d*
$CuFeS_2$	25.94	563	3.4311
$CuFeS_2$	29.75	943	3.0006
$CuFeS_2$	29.86	2960	2.9895
$CuFeS_2$	49.55	935	1.8379
$CuFeS_2$	58.31	854	1.5811

Table 2: The X-ray diffraction of *Shodhita Swarna Makshika*

Compounds	Two theta	Intensity	Value of *d*
Cu_2S	24.42	428	3.6418
	42.66	475	2.1174
	64.26	504	1.4483
Fe_3O_4	35.88	713	2.5002
	62.67	545	1.4810
FeS_2	33.42	731	2.6789
	40.18	419	2.2425
$FeSO_4$	18.49	482	4.7943
	54.31	561	1.6874
SiO_2	24.93	440	3.5687
	38.93	458	2.3111

Table 3: The X-ray diffraction of *Swarna Makshika Bhasma* (MB_1)

Compounds	Two theta	Intensity	Value of *d*
Fe_3O_4	31.48	588	2.8212
	36.24	1013	2.4764
	58.12	491	1.5857
CuS	30.37	510	2.9406
	62.84	538	1.4774
$FeSO_4$	18.97	445	4.7182
	43.6	498	2.0713
	55.69	512	1.690

Table 4: The X-ray diffraction of *Swarna Makshika Bhasma* (MB_2)

Compounds	Two theta	Intensity	Value of *d*
Fe_3O_4	31.48	532	2.8393
	35.86	982	2.5015
	57.46	507	1.6024
CuS	26.70	665	3.3360
	30.44	597	2.9335
	63.03	524	1.4697
$FeSO_4$	26.60	468	3.2635
	43.6	498	2.0713
	56.20	462	1.6031

Table 5: The X-ray diffraction of *Swarna Makshika Bhasma* (MB_3)

Compounds	Two theta	Intensity	Value of *d*
Fe_3O_4	35.85	906	2.5028
	57.63	489	1.5980
	63.11	489	1.5980
Cu_2O	31.38	562	2.8476
	40.55	476	2.2225
	53.98	447	1.7643
$FeSO_4$	18.52	507	4.7847
	26.66	651	3.3407
Fe_2O_3	38.93	493	2.3111

Table 6: The X-ray diffraction of *Kupipakwa Swarna Makshika Bhasma* (MB_4K)

Compounds	Two theta	Intensity	Value of *d*
$CuFeS_2$	29.46	1494	3.0287
	29.54	984	3.0212
	49.16	652	1.8518
	57.91	491	1.5909
Fe_3O_4	33.94	293	2.5018
	57.84	391	1.5912
	62.58	306	1.4071

Table 7: The X-ray diffraction of *Swarna Makshika Bhasma* (MB_4)

Compounds	Two theta	Intensity	Value of *d*
Fe_2O_3	35.78	506	2.5079
	57.75	344	1.5952
	62.56	346	1.4834
	65.84	336	1.4173
Cu_2O	31.27	311	2.8577
	35.45	424	2.7234
	40.40	322	2.2304
$FeSO_4$	26.66	315	3.3407
	44.64	400	1.9043
	56.35	319	1.7564
Cu_2S	30.409	312	2.9371
	43.36	318	2.0850
	48.18	288	1.9870

Conclusion

Particular benefit of diffraction analysis is that it discloses the presence of substances as that actually exists in the sample. This technique helps to identify and characterize the Ayurvedic raw material of mineral/metal origin and their processed form. XRD of different samples of *Swarna Makshika Bhasma* after comparing with JCPDS data revealed that raw *Swarna Makshika* contains $CuFeS_2$, which was converted into sulfides of copper and iron and oxide and sulfate of iron after *Shodhana*. Major compounds identified in *Bhasma* of different samples were Fe_3O_4, Fe_2O_3, FeS_2, $FeSO_4$, and Cu_2S. In *Bhasma* prepared by *Kupipaka* followed by *Putapaka*, Strongest peak of Fe_2O_3, Cu_2O and $FeSO_4$ were mainly identified.

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Conflicts of interest
There are no conflicts of interest.

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हिन्दी सारांश

विभिन्न विधियों से निर्मित स्वर्णमाक्षिक भस्म का एक्स–रे डिफ्रेक्शन द्वारा मानकीकरण

रमेशकुमार गुप्ता, विजय लक्ष्मी, चन्द्र भूषन झा

खनिजों व धातुओं का शोधन व मारण एक जटिल रासायनिक प्रक्रिया है जिसके द्वारा उनके अनैच्छिक व हानिकारक प्रभावों को दूर कर शरीर के लिए उपयोगी बनाया जाता है। रासायनिक विश्लेषणात्मक अध्ययन के द्वारा शोधन व मारण के पश्चात् द्रव्यों में हुए संरचनात्मक व रासायनिक परिवर्तनों का ज्ञान होता है। जिससे विभिन्न रस–ग्रन्थों में वर्णित विभिन्न संस्कारों के गुणों के रहस्यों को समझने में आसानी होती है। एक्स–रे डिफ्रेक्शन अध्ययन द्वारा निर्मित भस्मों के विभिन्न चरणों में हुए रासायनिक परिवर्तनों को समझा जा सकता है। इस अध्ययन में विभिन्न विधियों से निर्मित माक्षिक भस्मों का एक्स–रे डिफ्रेक्शन किया गया और यह पाया गया कि प्रत्येक भस्म में आयरन ऑक्साइड, कॉपर ऑक्साइड, कॉपर सल्फाइड, आयरन सल्फाइड, आयरन सल्फेट, आदि मुख्य रूप से विद्यमान थे।