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Comparative study of marketed samples of *Svarnamakshika Bhasma* an Ayurvedic medicinal preparation, using XRF and XRD Analysis

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ABSTRACT

Svarnamakshika Bhasma is an Ayurvedic medicinal preparation widely used by Ayurvedic practitioners for the management of various ailments. *Svarnamakshika Bhasma* needs to be prepared from *Svarnamakshika*, a mineral product identified and defined as Chalcopyrite (CuFeS2) in Ayurvedic Pharmacopeia of India (API). Minimum quantities of Iron, Copper and Sulphur, the three major elements necessary to be present in the *Svarnamakshika* (chalcopyrite), are prescribed as Pharmacopeial quality standard in the API monograph. Since included in the API, it is mandatory for the manufacturer to use *Svarnamakshika* (chalcopyrite) as specified in the API for preparation of *Svarnamakshika Bhasma*. However, it is generally observed that *Svarnamakshika* (chalcopyrite) complying with the standards prescribed by API is rarely found used for preparation of *Svarnamakshika Bhasma*. On this background seven samples of *Svarnamakshika Bhasma* comprising five marketed and two prepared by research scholars along with two samples of raw *Svarnamakshika* collected from research scholars were analysed in this study by using XRF and XRD analysis. The study indicates that only one marketed sample appears to have been prepared using *Svarnamakshika* (chalcopyrite) of pharmacopeial standard prescribed by API.

Key words: *Svarnamakshika, Bhasma, Chalcopyrite, X-Ray Fluorescence, X-Ray diffraction*

INTRODUCTION

Svarnamakshika Bhasma (SMB) is an Ayurvedic medicinal preparation widely used by Ayurvedic practitioners. *Svarnamakshika* is defined as a Copper ore containing Chalcopyrite (CuFeS2) mineral in Ayurvedic Pharmacopeia of India (API) Part I, Vol VII.[1] Chalcopyrite is a Copper Iron Sulfide, the most abundant Copper ore. Copper, Iron and Sulphur are the three major elements found in Chaclopyrite. API prescribes 5%, 20% and 12% as minimum contents of Copper, Iron and Sulphur respectively, in *Svarnamakshika* (chalcopyrite) as quality standard.[1] A processed *Svarnamakshika* (chalcopyrite) referred as *Sandrita Svarnamakshika* (Copper concentrate), defined as fine powder of Copper concentrate, in which minimum content Copper 12%, Iron 23% and Sulphur 28% has been set in API.[2] *Sandrcta Svarnamakshika* (Copper concentrate) retains overall minerology and other properties after processing the source mineral *Svarnamakshika* (chalcopyrite). The only difference is increase in its Copper content to more than 12%. Both *Svarnamakshika* (chalcopyrite) and *Svarnamakshika Sandrita* (Copper concentrate) can be used to prepare *Svarnamakshika Bhasma* (SMB) as per API. However, many research scholars have reported much higher content of Copper, Iron and Sulphur in Chalcopyrite found in mines across the globe. According to Haldar SK[3] Chalcopyrite is a Copper-Iron Sulfide mineral which in the purest form contains 34.5% Cu, 30.5% Fe, and 35.0% S. Shuming Wen et al[4] have reported high purity Chalcopyrite

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from Copper mine in China. On analysis, it was found to contain Copper 33.24%, Iron 27.22% and Sulphur 34.81%.

Three samples of Chalcopyrite obtained from Chambishi Copper Mine of Zambia, Hunan Province of China and Guangxi Province of China, were analysed by Hongbo Zhao et al.[5] XRD analysis of these three samples indicated Copper content as 35.8%, 31.9% and 30.1% respectively. Whereas, Iron content was 27.3%, 26.9% and 26.7%. Their Sulphur content was 29.4%, 28.6% and 27.6% respectively. Copper, Iron and Sulphur content in purest form of Chalcopyrite as reported by different research scholars as against their content prescribed in API is shown in Table 1.

Table 1: Copper, Iron and Sulphur content of various samples of Chalcopyrite

Reported by	Copper %	Iron %	Sulphur %
Shuming Wen et al	33.24	27.22	34.81
S.K. Haldar et al	34.5	30.5	35
Hongbo Zhao et al A	35.8	27.3	29.4
Hongbo Zhao et al B	31.9	26.9	28.6
Hongbo Zhao et al C	30.1	26.7	27.6
Mohapatra & Jha	33.8	28.60	32.85
Nambiar S et al	25.45	45.17	28.935
A P I Chalcopyrite	Not < 5	Not < 20	Not < 12
API Copper Concentrate	Not < 12	Not < 23	Not < 28

It is essential to ensure presence of major ingredients in quantities prescribed by API in the drug. Moreover, as pe rule 168 of Drugs and Cosmetics Act and Rules 1945[6] thereunder, compliance of standards of identity, purity and strength as given in the editions of API, is mandatory for manufacturing Ayurvedic drugs described in API for sale and distribution. Considering the significance of this rule and therapeutic significance of chemical composition of a drug, it was decided to collect samples of *Svarnamakshika* (chalcopyrite) with the marketed drug *Svarnamakshika Bhasma* (SMB), and analyse the content of the major ingredients Iron, Copper and Sulphur present in them. The raw material *Svarnamakshika* (chalcopyrite) used to prepare *Svarnamakshika Bhasma* could not be obtained from the manufacturers. However, five samples of *Svarnamakshika Bahsma* were purchased and collected directly from the market. In addition, two samples each claimed to be *Svarnamkashika* and *Svarnamakshika* Bhasma were also collected from research scholars. All these collected samples were subjected to analysis using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD). The results indicate a wide variation in quantities of major ingredients Iron, Copper and Sulphur present in the samples of *Svarnamakshika Bhasma*. Analysis of the raw material claimed to be *Svarnamakshika* collected from the researchers also showed that they didn't comply with the standards prescribed by API. This was particularly evident with regard to the quantity of Copper. Copper was found markedly absent in all except one marketed sample of *Svarnamakshika Bhasma* and the one collected from research scholar. The study underlines the need of analysis of raw materials and the marketed Ayurvedic medicinal products with respect to identity, purity and strength.

Materials and Methods

Seven samples of *Svaranamakshika Bhasma* comprising five marketed and two prepared by research scholars, were collected for this study. In addition, two samples claimed to be *Svarnamakshika* used by the researchers to prepare *Svarnamakshika Bhasma* were also collected. All the samples were subjected to XRF and XRD analysis with an object of assessing product uniformity of *Svarnamakshika Bhasma* with respect to chemical composition.

X Ray Fluorescence Analyser

Vanta handheld X Ray Fluorescence Analyzer was used for XRF analysis in this study. It is an energy dispersive X-ray fluorescence spectrophotometer used to perform identification and analysis of elements contained in the test sample. This method identifies the elements in a substance and quantifies them. An

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element is defined by its characteristic X-ray emission energy (E). The amount of element present is determined by measuring the intensity of its characteristic line. In XRF spectrometry, primary X-ray photons are emitted from a (source) X-ray tube and strike the sample. These primary photons knock electrons out of the innermost orbitals vacating a space. An electron from outer orbital moving into this vacant space in the inner orbit, emits an energy known as secondary X-ray photon. The secondary X-ray photon is characteristic of a specific element.

Method

The sample powder filled in the sleeved sample cup covered with film was subjected to XRF analysis using Vanta XRF analyser. Positioning the measurement window of the analyser directly over the surface of the film, the test was initiated. The results displayed on the analyser screen were recorded at the completion of the test.

X Ray Diffraction

XRD analysis is a non-destructive analytical technique. A monochromatic X-ray beam is used to irradiate a sample in this technique. The X-ray source and detector are positioned at variable angles around the sample, which is usually flat. X-ray diffractogram obtained in the analysis is used to identify the crystallographic structure and chemical composition of the sample. X-Powder software is used for interpreting the X-ray diffractogram for the analysis of natural minerals, artificial compounds, biological crystals and other solid materials with the help of database. It is used for identification, quantification and characterization of the crystalline components of solid samples.

Method

Terra-II portable X-ray Diffraction analyser was used for XRD analysis of the samples in the present study. Analysis was done using SwiftMin software connected to the analyser. A diffractogram obtained was subjected to analysis using X- powder software. Peaks in the diffractogram related to specific chemical compounds were identified with the help of the database.

Results

Chalcopyrite being the source material of *Svarnamakshika Bhasma* (SMB), XRF analysis of the samples was carried out to determine quantity of three major elements Iron, Copper and Sulphur. Quantity of Iron in *Svarnamakshika Bhasma* (SMB) varied from 17.62% (SMB-ADT) to 63.26% (SMB-SUD) in the analysed samples. Whereas, Copper content varied from 349 ppm (SMB-BTR) to 16.08% (SMB-DTP). Quantity of Sulphur varied from 2540 ppm (SMB-BDT) to 4.728% (SMB-AKL). In addition to Iron, Copper and Sulphur; Aluminium, Calcium and Silica were also detected in significant quantities in some of the samples. Along with these elements significant quantities of light elements (LE) were detected in all the samples. Composite content of light elements in the samples was observed quite high ranging from 28.95% (SMB-SUD) to 64.92%. (SMB-ADT). (Table 2)

Table 2: Elements detected in XRF analysis of *Svarnamakshika Bhasma* (SMB)

	SMB - SUD	SMB DTP	SMB KNG	SMB BDT	SMB AKL	SMB BTR	SMB ADT
Fe	63.26 %	33.82 %	42.82 %	24.03 %	49.28 %	56.3%	17.62 %
Cu	595 ppm	16.08 %	2650 ppm	779 ppm	1346 ppm	349 ppm	3.521 %
S	8520 ppm	4.513 %	4.728 %	2540 ppm	4.577 %	1.584 %	-
LE	28.95 %	42.91 %	46.79 %	59.42 %	38.12 %	36.18 %	64.92 %
Si	1.4 %	8560 ppm	1.470 %	7.30%	1.522 %	2.157 %	9.18%
Al	1.38%	7800 ppm	1.03%	4.30%	9100 ppm	1.48%	1.62%
Ca	-	4050 ppm	1.957 %	2.001 %	4.420 %	1.416 %	2.232 %

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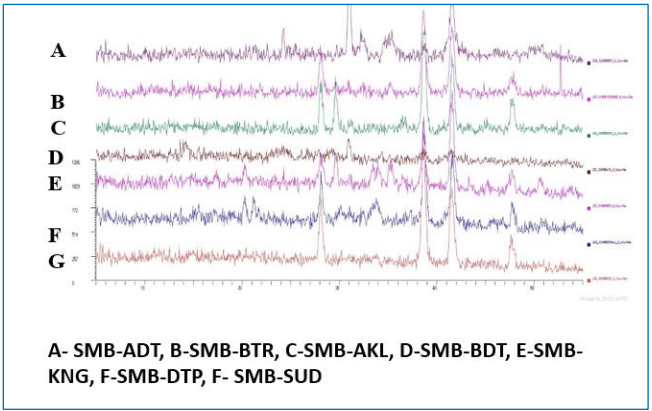
XRD Analysis

Figure 1: X- Ray Diffractograms of seven samples of *Svarnamakhika Bhasma* (SMB)



A- SMB-ADT, B-SMB-BTR, C-SMB-AKL, D-SMB-BDT, E-SMB-KNG, F-SMB-DTP, F- SMB-SUD

Table 3: Compounds detected in XRD of *Svarnamakshika Bhasma* - SMB-SUD and SMB-AKL

***Svarnamakshika Bhasma* - SMB-SUD**			***Svarnamakshika Bhasma* - SMB-AKL**		
Compound	**2 Theta**	**d spacing**	**Compound**	**2 Theta**	**d spacing**
Iron Oxide Fe2 O3	28.087 38.682 41.558	3.6862 2.7008 2.5213	Iron Oxide Fe2 O3	28.115 38.654 41.614	3.6826 2.7027 2.5181
			Potassium Iron Sulfate Hydrate Fe H8 K2 O12 S	16,884 30.189 54.172	6,0928 3.4348 1.9645

Table 4: Compounds detected in XRD of *Svarnamakshika Bhasma* - SMB-BDT and SMB-KNG

***Svarnamakhika Bhasma* SMB-BDT**			***Svarnamakhika Bhasma* SMB- KNG**		
Compound	**2 Theta**	**d spacing**	**Compound**	**2 Theta**	**d spacing**
1. Iron Oxide Acetate Hydroxide Hydrate	8.060 26.676 46.676	12.7276 3.8773 2.25790	Iron Oxide Fe2 O3	28.115 38.709 41.558	3.6826 2.6990 2.5213
C2 H6 Fe2 O6 H2 O					
2. Iron Sulfate Hydrate Fe S O4 (H2 O)4	18.793 25,957 55.223	5.4788 3.9828 1.9299	Gamma ·Iron Oxide Fe21.333 O32	35.362 41.697 50.963	2.9451 2.5133 2.0791
3. Iron Sulfate Hydrate Fe2 (S O4)3 (H2 O)9	11.573 22.389 45.791	8.8719 4.6075 2.2992	Potassium Oxonium Iron Sulfate Hydroxide Fe3 H6 K O14 S2	20.287 33.813 53.619	5.0791 3.0758 1.9832
4. Iron Hydrogen Sulfate Hydrate Fe2 H6 O18 S4	13.343 31.019 52.623	7.6991 3.3451 2.0180	4. Sodium Iron Sulfate Hydroxide Fe3 H6 Na O14 S2	20.370 33.951 53.757	5.0586 3.0637 1.9785
5. Iron Chloride Hydrate 2 Fe Cl3 !7 H2 O	15.944 23.717 55.278	6.4497 4.3529 1.9282	5. Sodium Oxonium Iron Sulfate Hydroxide Fe3 H7.26 Na0.58 O14.42 S2	20.508 33.868 53.674	5.0249 3.0709 1.9813

Table 5: Compounds detected in XRD of *Svarnamakshika Bhasma* - SMB-DTP

***Svarnamakhika Bhasma* SMB-DTP**		
Compound	**2 Theta**	**d spacing**
Iron Oxide Fe2 O3	28.115 38.737 41.614	3.6826 2.6971 2.5181
Calcium Iron Oxide Ca2 Fe O3.5	14.975 40.978 54.449	6.8641 2.5555 1.9553
Potassium Iron Sulfate Hydroxide K Fe3 (SO42 (O H)6	20.287 33.813 53.923	5.0791 3.0758 1.9729

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Potassium Sodium Iron Sulfate Hydrate K6.8 Na5.96 (H3 O).8 Fe6.05 O2 (S O4)12 (H2O)	5.681 13.509 37.437	18.0497 7.6049 2.7873
Sodium Iron Sulfate Na2 Fe (S O4)2	17.963 33.537 37.299	5.7297 3.1005 2.7972
Sodium Oxonium Iron Sulfate Hydroxide Na0.58 (H3 O)0.42 Fe3 (S O4)2 (OH)6	20.480 33.896 53.730	5.0316 3.0685 1.9794
Copper Iron Sulfate Hydrate Cu0.47 Fe0.53 (SO4) (H2O)	21.697 29.858 33.675	4,7525 3.4721 3.0881
Sodium Copper Sulfate Hydroxide Hydrate Cu2 H3 Na O10 S2	15.556 30.023 37.299	6.6093 3.4534 2.7972

Table 6: Compounds detected in XRD of *Svarnamakshika Bhasma* - SMB-BTR and SMB-ADT

***Svarnamakshika Bhasma* - SMB-BTR**			***Svarnamakshika Bhasma* - SMB-ADT**		
Compound	**2 Theta**	**d spacing**	**Compound**	**2 Theta**	**d spacing**
Iron Oxide Fe2 O3	28.032 38.737 41.586	3.6862 2.6971 2.5197	Iron Oxide Fe3O4	21.282 35.086 41.420	4.8440 2.9676 2.5294
Potassium Iron Sulfate Hydrate Fe H8 K2 O12 S	15.446 34.643 38.848	6.6563 3.0043 2.6898	Iron Oxide Hydroxide Fe H O2	20.840 35.445 55.279	4.9457 2.9384 1.9282
			Copper Iron Phosphate Cu2 Fe5 (P O4)6	15.086 34.892 55.306	6.8140 2.9835 1.9273

Elements detected in XRF analysis were used as the basis for finding out presence of their compounds in the XRD analysis. Complex compounds especially Oxides and Sulfates of Iron, Copper and light elements like Sodium, Potassium and Calcium were detected in almost all samples of *Svarnamakshika Bhasma* (SMB). (Fig 1 and Table 3 to 6).

Oxides

Iron oxide Fe2O3 was observed to be the principal compound in all the samples of *Svarnamakshika Bhasma* (SMB) except in SMB-BDT. However, Iron Oxide Acetate Hydroxide Hydrate Fe2 O6! C2 H6 H2 O was detected in it (Figure 1, Table 4). Other variants of oxide of Iron like Gamma Iron oxide (Fe21.333 O32) and Iron oxide Hydroxide (Fe H O2) were detected respectively in SMB-KNG (Figure 1 Table 4) and SMB-ADT (Figure 1, Table 6). Presence of Calcium Iron Oxide (Ca2 Fe O3.5) was observed in SMB – DTP. (Figure 2, Table 5).

Sulfates

Sulphur is a major content of *Svarnamakshika* (chalcopyrite). Sulfates of Iron like Iron Sulfate Hydrate (Fe SO4 (H2 O)), Fe2 (SO4)3 (H2O)9 and Iron Hydrogen Sulfate Hydrate (Fe2 H6 O18 S4) were found present in SMB-BDT (Table 4). Sulfates of Iron with Potassium and Sodium like Potassium Iron Sulfate Hydroxide (K Fe3 (SO4)2 (O H)6 and Potassium Sodium Iron Sulfate Hydrate K6.8 Na5.96 (H3 O).8 Fe6.05 O2 (S O4)12 (H2O) were also detected in SMB-KNG (Table 4). Whereas, Potassium Iron Sulfate Hydrate Fe H8 K2 O12 S was detected in SMB-BTR (Table 6).

Compound of Iron and Sodium, Sodium Oxonium Iron Sulfate Hydroxide (Fe3 2 (OH)6 (SO4) Na 0.58 (H3O) 0.42) was detected in SMB-DTP and SMB-KNG. Whereas Sodium Iron Sulfate (Na2 Fe (S O4)2) and Sodium Iron Sulfate Hydroxide (Fe3 H6 Na O14 S2 were found present respectively in SMB-DTP (Table 5) and SMB-KNG (Table 4).

Copper was detected in two samples SMB-DTP (16.08%) (Table 5) and SMB-ADT (3.521%) in XRF analysis (Table 6). Therefore, these two samples were specifically analysed for detecting the presence of compounds of Iron and Copper. Copper Iron Sulfate Hydrate (Cu0.47 Fe0.53 (SO4) (H2O) and Sodium Copper Sulfate Hydroxide Hydrate (Cu2 H3 O10 S) were found present in SMB DTP (Table 5). Whereas Copper Iron Phosphate (Cu2 Fe5 (PO4)6) was detected in SMB-ADT (Table 6).

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Raw *Svarnamakshika* and *Svarnamakshika Bhasma*

Table 7: Element content in Raw *Svarnamakshika* (RSM) and *Svarnamakshika Bhasma* (SMB)

	RSM-BTR	SMB-BTR	RSM-ADT	SMB-ADT	API standard
Fe	41.18%	56.3%	33.21%	17.62%	Not less than 20%
Cu	140 ppm	349 ppm	3.844%	3.521%	Not less than 5%
S	33.47%	1.584%	8.47%	-	Not less than 12%
LE	23.26%	36.18%	45.80%	64.92%	

Two samples of raw *Svarnamakshika* were analysed in this study. Both these samples were collected from the research scholars. None of the two complied with the API prescribed quality standard of Copper and Sulphur content. RSM-ADT contained 3.844% copper. Whereas Copper content in RSM-BTR was a meagre 349 ppm. Sulphur content in RSM-BTR was 33.47%, much higher than prescribed. Whereas, it was only 8.47% in RSM-ADT, which was much below the prescribed quantity of not less than 12%. Iron was found in the measure of 41.18% and 33.21% respectively in RSM-BTR and RSM ADT in XRF analysis (Table 7).

XRD Analysis of Raw *Svarnamakshika*

Figure 2: X Ray Diffractogram of Raw *Svarnamakshika* (RSM- BTR) and *Svarnamakshika Bhasma* (SMB- BTR)

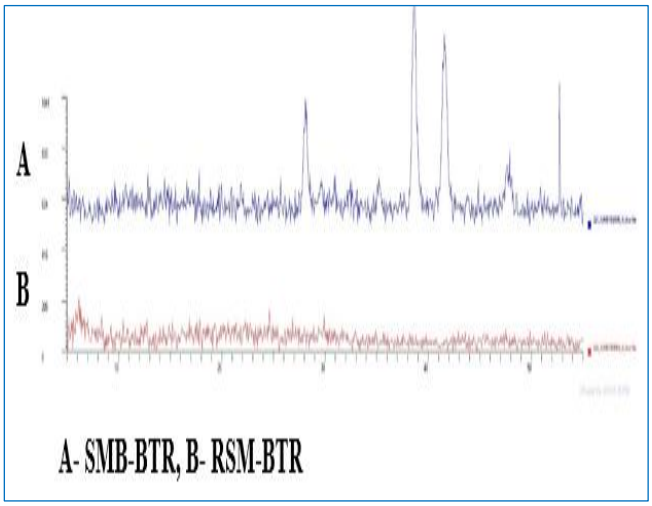

Figure 3: Compounds detected in XRD of Raw *Svarnamakshika* RSM- ADT and *Svarnamakshika Bhasma* (SMB- ADT)

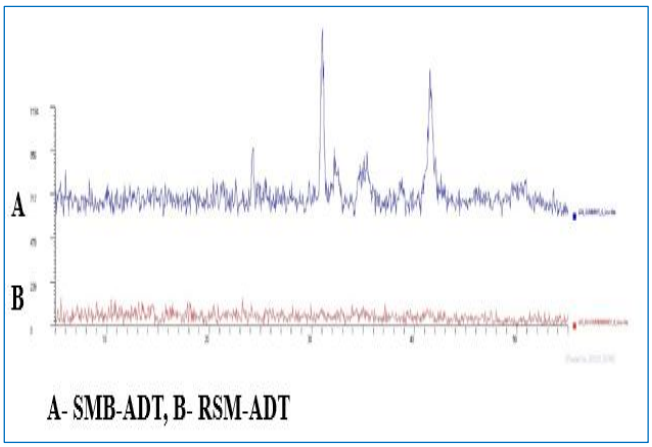

Table 8: Compounds detected in XRD of Raw *Svarnamakshika* - RSM-BTR and RSM-ADT

Raw *Svarnamakshika* - RSM-BTR			Raw *Svarnamakshika* - RSM-ADT		
Compound	**2 Theta**	**d spacing**	**Compound**	**2 Theta**	**d spacing**
Ammonium Iron Sulfate Hydrate Fe2 H16 N2 O20 S ·	14.865 27.267 39.207	6.9149 3.7962 2.65660	Iron Sulfate Fe6 S8 O33	17.742 28.281 39.899	5.8005 3.6614 2.6217
Sodium Iron Sulfite Hydrate Fe H4 Na5 O14 S4	10.439 21.310 35.169	9.8326 4.8378 2.9608	Copper Iron Sulfide Cu4 Fe5 S	20.314 23.271 33.813	5.0723 4,4345 3.0758
Potassium Iron Sulfate Hydrate Fe H8 K2 O12 S	21.061 32.098 51.129	4.8943 3.2355 2.0728	Potassium Sodium Oxonium Iron Sulfate Oxide Hydrate Fe6 H36.23 K3.5 Na4.59 O67.16	11.435 34.062 52.259	8.9572 3.0540 1.9956

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Calcium Iron Sulfate Hydroxide Hydrate Ca6 Fe2 H64 O50 S3	10.605 29.913 54.338	9.6792 3.4659 1.9589	Potassium Sodium Iron Sulfate Hydrate Fe7 H36 K2 Na6 O68 S12	5.663 27.562 48.031	18.380 3.7551 2.1978
			Calcium Iron Oxide Iodide Hydrate Ca4 Fe2 H13 I2 O12.5	13.343 26.925 55.168	7.6991 3,8421 1,9317

XRD analysis indicated that RSM-BTR contained various forms of Sulfates of Iron. Whereas, RSM-ADT contained Iron sulfate (Fe6 S8 O33) along with Sulfide and Oxide compounds of Copper and Iron. (Figure 2 and 3 and Table 8).

Discussion

Svarnamakshika Bhasma (SMB) needs to be prepared from *Svarnamakshika*, a mineral product identified and defined as Chalcopyrite (CuFeS2) in API.[1] Since included in the API, it is mandatory on the part of manufacturer to use *Svarnamakshika* (chalcopyrite) as specified in the monograph for preparation of *Svarnamakshika Bhasma* (SMB). However, it is observed that *Svarnamakshika* (chalcopyrite) as specified in API is rarely used to prepare *Svarnamakshika Bhasma* (SMB). Considering the significance of the issue, the present study was planned and designed accordingly. Iron, Copper and Sulphur are the three major elements present in *Svarnamakshika* (chalcopyrite). Minimum quantities of these three elements necessary to be present in the *Svarnamakshika* (chalcopyrite), are prescribed as Pharmacopeial quality standard in the API monograph.[1] Interestingly, the minimum content of Copper, Iron and Sulphur in *Svarnamakshika* (chalcopyrite), prescribed in API is much lower as compared to the contents of these elements in high purity chalcopyrite as reported by various research scholars[3.4.5] (Table 1). The present study was designed to determine the quantities of these three elements in the *Svarnamakshika* used to prepare the *Svarnamakshika Bhasma* (SMB).

XRF Analysis

Iron content in the two samples of raw *Svaranamakshika* (RSM) collected from the research scholars was 41% (RSM -BTR) and 33.21% (RSM- ADT) (Table 7). In this regard, both these samples comply with prescribed API pharmacopeial standard of not less than 20% (Table 7). However, the two samples failed to comply with Copper content of not less than 5% as prescribed by API. Copper content of RSM- ADT was 3.844%. Whereas, Copper was almost absent (140 ppm) in *Svarnamakshika* (RSM-BTR) (Table 7). It clearly indicates that RSM-BTR is not the *Svarnamakshika* (chalcopyrite) as defined in the monograph in API. The sample RSM-ADT also failed to qualify the standard of Sulphur content. Thus, although RSM-ADT did contain Iron, Copper and Sulphur, the Copper and Sulphur content in it was much below the requirement of API standard.

The XRF analysis of *Svarnamakshika Bhasma* (SMB) showed that Copper is present in only one marketed sample (SMB-DTP- 16.08%.) (Table 2) and in one sample collected from research scholar (SMB-ADT – 3.521%) (Table 7). Copper content in other samples of SMB was in ppm measure. The observation indicates that only one marketed sample of *Svarnamakshika Bhasma* - SMB- DTP was likely to be prepared from *Svarnamakshika* (chalcopyrite) complying with the API prescribed pharmacopeial standard with respect to Copper content. Whereas no other marketed samples tested in the present study appeared prepared from *Svarnamakshika* (chalcopyrite) as defined in the API monograph.

Wide variation in Iron content in the samples of *Svarnamakshika Bhasma* (SMB) was observed in this study. It ranged from 17.62% (SMB-ADT) to 63.26% (SMB-SUD). (Table 2). The third major ingredient of *Svaranmakshika* (chalcopyrite) is Sulphur. Raw *Svarnamakshika* (RSM) used by the manufacturer was not available for the study, hence could not be tested.

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However, samples of marketed *Svarnamakshika Bhasama* (SMB) were tested for presence of Sulphur. It was observed that 4.728% of Sulphur was detected in SMB-KNG, which was the highest among all tested samples. Whereas, it was completely absent in one sample of SMB -ADT (Table 2). Sulphur content in RSM-BTR and RSM-ADT was 33.47% and 8.47% as against not less than 12% prescribed in API monograph (Table 7). The process of preparation of *Svarnamakshika Bhasma* (SMB) is a type of oxidation process. As a result, most of the Sulphur combining with Iron and Copper, forms their Sulfates and Sulfides, during the process of preparation of *Svarnamakshika Bhasma*. Differing content of Iron, Copper and Sulphur in *Svarnamakshika Bhasma* (SMB) have been reported by number of workers in the past.

Cu 0.337%, Fe 54.29% S 0.63% in SMB has been reported by means of FESEM E DAX analysis by Bharadwaj R et al.[7] Mohapatra and Jha[8] have reported Cu-33.08%, Fe-28.60% and S-32.85% in raw *Svarnamakshika* (Table 1) and Cu-29.40%, Fe-32.26%, S-02.45% in *Svarnamakshika Bhasma* in their EDAX analysis.

Nambiar S. et.al.[9] have reported presence of Cu 25.45%, Fe 45.17% and S 28.935 % in raw *Svarnamakshika* (Table 1). They have prepared *Svarnamakshika Bhasma* by two different methods. Iron content 27.49% and 30.27%, Copper content 6.855% and 6.24% with Sulphur 4.45% and 2.41%; was respectively reported in EDAX studies of *Svarnamakshiaka Bhasma* prepared by the two methods. Interestingly, the Iron and Copper content along with Sulphur content appears significantly reduced in *Svarnamakshaika Bahsma* as compared to the raw *Svarnamakshika* in this study. Reduction in Sulphur content due to oxidation during the process is justified. However, reduction in Iron and Copper content during processing remains to be answered by the worker in this study. Loss of these two elements is likely to be a result of manual handling of the material. Another important ingredient of the *Svarnamakshika Bhasma* is formed by light elements. They mainly comprise of Oxygen and Hydrogen. The content of light elements ranges from 28.95% in SMB-SUD to 64.92% in SMB-ADT. Apart from these elements, Aluminium (Al), Calcium (Ca) and Silica (Si) were also detected in minor quantities in almost all the samples of SMB. (Table 2).

The study indicates that although highly pure Chalcopyrite is rich in Copper and Iron content, it is not used by any manufacturer to prepare *Svarnamakhsika Bhasma*. Use of Chalcopyrite much lower in Copper and Iron content is permitted by API, a legal document mandatory to be followed by manufacturers of Ayurvedic Medicine in India. Basically *Svarnamakshika* (chalcopyrite) being a mineral, is composed of complex compounds. The major element Copper, along with Sodium, Potassium, Calcium, and Ammonium combining with Sulphur form their complex Sulfides and Sulfates as a result of speciation. Such complex compounds are also found in *Svarnamakshika Bhasma*.

XRD Analysis

XRD analysis is the most important analysis in determining the chemical composition of *Bhasma* preparations. In the present study, XRD analysis revealed the complex chemical composition of *Svarnamakshika Bhasma*, which differed widely from sample to sample. Iron oxide (Fe2O3) was observed present in all the samples of *Svarnamakshika Bhasma* except in one sample (SMB- BDT). Presence of Fe2O3 in *Svarnamakshika Bhasma* has been reported by different workers in the past. Nair RR et al[10] have reported Fe2O3- 75.54% during XRF analysis in their study. Bharadwaj R et al[7] and Mohapatra and Jha[8] also report presence of Fe2O3 in *Svarnamakshika Bhasma* analysed by them.

Apart from Fe2O3 other forms of oxides of Iron have been detected in *Svarnamakshika Bhasma* in the present study. In SMB-ADT Iron oxide hydroxide (Fe2O6 C2H6 H2O) (Table 6) was found present. In SMB-DTP along with Iron oxide, Calcium Iron oxide (Ca2 FeO3.5) was also detected (Table 5).

In addition to oxides, various Sulfate compounds like Sulfate of Iron, Iron and Potassium, Iron and Sodium, and Iron and Copper; were detected in different tested samples of *Svarnamakshika Bhasma*. Iron Sulfate

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Hydrate (FeSO4 (H2O)4) and Iron Hydrogen Sulfate Hydrate (Fe2 H6 O18 S4) were found present in one sample SMB-BDT (Table 4).

Sulfates of Iron and Potassium

Potassium Oxonium Iron Sulfate Hydroxide (K Fe3 (SO4)2 (OH)6, Sodium Oxonium Iron Sulfate Hydroxide (Fe3 H7.26 Na 0.58 O14.42 S2) and Sodium Iron Sulfate Hydroxide (Fe3 H6 Na O14 S2) were observed present in one sample (SMB-KNG) (Table 4). Whereas Potassium Iron Sulfate Hydrate (Fe H8 K2 O12 S) was found present in SMB-BTR. (Table 6).

Sulfates of Iron and Sodium

Sulfates of Iron and Sodium were found present in two samples SMB DTP and SMB -KNG. Sodium Oxonium Iron Sulfate Hydroxide (Fe3.2 (OH)6 (SO4) Na0.58 (H3O) 0.42) and Sodium Iron Sulfate (Na2 Fe (SO4) 2) were found present in SMB-DTP. (Table 5). Whereas, Sodium Oxonium Iron Sulfate Hydroxide (Fe3.2 (OH)6 (SO4) Na0.58 (H3O) 0.42) along with Sodium Iron Sulfate Hydroxide (Fe3 H6 Na O14 S2) were detected in SMB -KNG (Table 4).

Sulfate compounds with both Iron and Copper

Iron and Copper compounds were found present in only two samples SMB-DTP and SMB-ADT in this study. The finding is in accordance with the observations noted in XRF analysis, as Copper was detected only in these two samples in XRF analysis. Copper Iron Sulfate Hydrate (Cu 0.47 Fe 0.53 (SO4) (H2O) and Sodium Copper Sulfate Hydroxide Hydrate (Cu2 H3 Na O10 S2) were detected in SMB-DTP (Table 5). Whereas, Copper Iron Phosphate was found in SMB-ADT (Table 6).

The two samples of *Svarnamakshika* collected from the research scholars were also subjected to XRD analysis. One Copper compound Copper Iron Sulfide (Cu4Fe5 S), with Iron Sulate (Fe6 S8 O33), Potassium Sodium Iron Sulfate Hydrate (Fe H 36 K2 Na 6 O 68 S12) and Potassium Sodium Oxonium Iron Sulfate Oxide Hydrate (Fe6 H36.23 K3.5 Na 4.59 O 67.16) along with Calcium Iron Oxide Hydrate (Fe 6 H 36.23 K 3.5 Na 4.59) were detected in RSM-ADT (Table 8). Whereas, Ammonium Iron Sulfate Hydrate (Fe 6 H 16 N 2 O 20 S), Sodium Iron Sulfite Hydrate (Fe H 4 Na 5 O14 S 4), Potassium Iron Sulfate Hydrate (Fe H 8 K2 O12 S) and Calcium Iron Sulfate Hydroxide Hydrate (Ca 6 Fe 2 H 6.54 O 50 S 3) were found present in RSM – BTR (Table 8). No copper compound was detected in RSM-BTR.

XRD analysis of the samples analysed in this study indicates that *Svarnamakshika*, a mineral, is composed of complex chemical compounds made of Iron, and when present of Copper as well. Both these metals form various compounds combining with Calcium, Potassium, Sodium, Ammonium, the elements found naturally mixed with almost all the minerals due to speciation. Copper compounds could be detected in only one marketed sample SMB-DTP in this study. Whereas, Iron oxide (Fe2O3) was found present in all the samples. The observation indicates that with exception of one manufacturer all other manufacturers whose *Svarnamakhsika Bhasma* was analysed in this study, do not use *Svarnamakshika* (chaclopyrite) as prescribed by API for manufacturing *Svarnamakshika Bhasma*. It is observed in this study that Ayurvedic *Bhasma* preparations are neither single chemical entities nor simple chemical compounds. They are all mixtures of complex chemical compounds. Apart from the chemical compounds of the main source metal, compounds of elements like Calcium, Sodium, Potassium, naturally found in minerals are also present in the *Bhasma* preparations.

This study has also raised a question regarding the quality of Ayurvedic *Bhasma* preparations sold in the market. The five marketed samples of *Svarnamakshika Bhasma* analysed in this study varied widely from each other in their chemical composition. Pharmacological action of a drug is closely dependent on its chemical composition. Uniformity in chemical composition of drug products produced in multiple batches is essential to ensure uniformity in their pharmacological action. Such uniformity appears completely missing in the five marketed samples of *Svarnamakshika Bhasma* analysed in this study. Absence of this uniformity is observed as a result of use raw *Svarnamakshika* not complying with pharmacopeial standards prescribed by API.

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Conclusion

Overall study indicates absence of uniformity in the marketed samples of *Svarnamakhika Bhasma* analysed in this study. Wide variation in chemical composition in the samples of *Svarnamakshika Bhasma* tested in this study with respect to individual elements and chemical compounds, was observed. Different varieties of Oxides and Sulfides and Sulfates of Iron, Sodium and Potassium combined with Iron were found formed in all samples of *Svarnamakshika Bhasma* due to speciation. Only two samples SMB-DTP and SMB-ADT contained Copper compounds. As both of them were prepared from Chalcopyrite (CuFeS2). It is also significant to note that although Chalcopyrite is principally a Copper ore, the minimum quantity of Copper content in the Chalcopyrite prescribed as a pharmacopeial quality standard by Ayurvedic Pharmacopeia of India (API) is much lower than the actual Copper content present in high purity Chalcopyrite as reported by many research scholars. The study indicates that out of five marketed samples tested, only one marketed sample SMB-DTP being composed of Iron and Copper compounds, can be termed as *Svarnamakshika Bhasma*. Whereas, all other marketed samples do not deserve to be referred as *Svarnamakshika Bhasma*. They all are Iron compounds, likely to be prepared from Iron pyrite, referred as *Vimal* in Ayurvedic classics.

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