ORIGINAL ARTICLE

Characterization of Rajata Bhasma (traditional calcined silver preparation)

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Abstract

Introduction: Rajata Bhasma (RB), a calcined preparation of silver, is widely used in various herbo-mineral compounds and ample used as a single drug with different Anupanas (adjuvant) in several diseases. It is potentially used in numerous herbo-mineral formulations such as Kasturi bhairav Rasa, Grahani kapata Rasa, Jayamangala Rasa, and Trilokya chintamani Rasa. **Objectives:** Several works emphasizing standard method of preparation has been established, but there are few works focusing analytical profile of RB, hence objectives were to establish an analytical profile of RB. **Materials and Methods:** RB was subjected for X-ray diffraction (XRD), X-ray fluorescence, and particle size analysis. **Results:** Mean particle size of RB was 1.04 µm, and XRD results revealed RB was Ag2Hg2S (imitrite) initially and was in Acanthite (Ag2S) form in the final product. **Conclusion:** RB was found to be in silver sulfide (Ag2S) having a considerable reduction in particle size. Final product was devoid of mercury and other heavy metal contamination.

Key words: Bhasma, Rajata, Silver, XRD, SEM-EDX, Characterization

INTRODUCTION

Rasashastra is a pharmaceutical science, dealing mainly with the processing and therapeutic utilization of metals, and minerals.[1] It emphasizes on conversion of metal and minerals into organometallic biocompatible form can easily be absorbed and assimilated.[2] Processed metals were used very frequently by seers of the Indian tradition in different disease conditions with great authority.[3] Bhasmas (calcined formulation) are intriguing formulations of metals such as Cu, Ag, Au, Fe, Zn, and Hg where these are associated with organic macromolecules derived from the herbal extract making these biologically assimilable.[4] These formulations of herbomineral origin have been reported for a higher concentration of heavy metals.[5] Ayurvedic formulations are banned in most of the countries in Europe.[6] The preparations of these Bhasmas involve several steps such as purification of metal/mineral followed by Bhavana (levigation) with different herbal juices and repeated calcination cycles till desired characteristics appear.[7] Several steps followed will ensure the formation of herbo-mineral complex compound formation. It is the form and of the drug which decides whether it is a poison or nectar, hence in the first place, it is need of hour to characterize and standardize the formulations. Characterization of several Dhatu (metal) Bhasmas have been attempted such as Swarna (gold),[8] Tamra (copper),[9,10] Loha (iron),[11,12] Naga (lead),[13-15] Vanga (tin),[16] and Yashada (zinc).[17] Standard manufacturing procedure of Rajata Bhasma (RB) has been attempted earlier,[18,19] instrumental neutron activation analysis of RB revealed 23.4% Ag besides As (14.2%), S (19.9%), P (5.14%) and Na (1.28%), with Mn (183 µg/g), and Au (140 ng/g) in traces suggesting the possibility of Ag_2S or As_2S_5.[4] In this study, characterization of RB by X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy (SEM), and particle size determination was carried out to establish analytical standards.

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MATERIALS AND METHODS

Brief Information Regarding Samples

RB was prepared as per classical guidelines.[20] Pharmaceutical standardization of the samples was previously published and the methods adapted was as follows; silver, mercury, and sulfur were subjected for Shodhana (purification). Purified silver and mercury were triturated to form amalgam in a mortar. Purified sulfur was added to the amalgam and triturated till the formation of fine powder. This was followed by impregnation with *Aloe vera* juice to form pellets. Dried pellets were placed in an earthen saucer and incinerated in *LaghuPuta* (Square pit 30 [l] 30 [b] 30 [h]) with cow dung cakes. For subsequent two incineration cycles, Kajjali (black sulfide of mercury) was added and triturated with *A. vera* juice. Repeated incineration cycles were adapted till fulfillment of Bhasma Siddhi Lakshana (desired characters of Bhasma). Thus, 17 incineration cycles were given till all the classical parameters such as Rekapurna (entering the furrows of the finger), Varitara (floating on the water surface), Nischandra (devoid of shine), Niswadu (devoid of metallic taste), Apunarbhava (do not regain metallic nature), and Niruttha (devoid of free metal) were fulfilled. Cow dung cakes required for each incineration cycle were 22-26 weighing 2.5 kg.[19]

Instrumental Analysis

Crystallinity of the Bhasma was studied using XRD (XRD, machine JEOL make, JPX 8). CU- Kα, λ = 1.5418 was the radiation used from NITK, Surathkal.

Particle Size of the sample was determined by the laser scattering particle size analyzer (LA-910) using triple distilled water as a medium in NITK, Surathkal.

The morphology of the samples was characterized using scanning electron microscope (SEM, JEOL make, model 6380LA). Elemental compositions at various regions in the coating were estimated using energy dispersive spectroscopy (EDS) attached to SEM in NITK, Surathkal.

Model S4 Pioneer Bruker aXS, XRF analysis was performed for RB from IIT, Madras.

RESULTS AND DISCUSSION

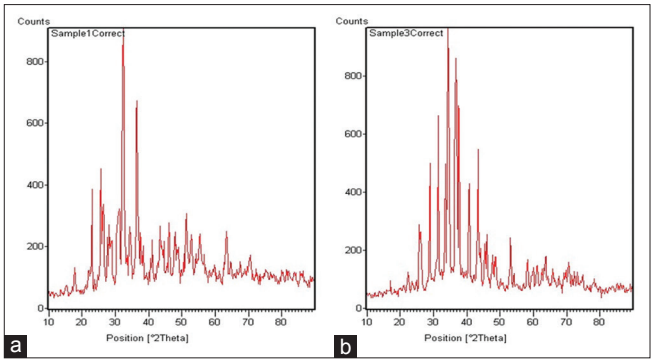
The advanced instrumental analysis of the Bhasma sample is extremely important to determine the chemical composition of the material to be used as the drug. Samples before and after processing were analyzed to determine the changes taking place in the sample with the various treatment procedures.

XRD report of intermediate product (RBK) and RB showing Ag_2Hg_2S and Ag_2S in major phase, i.e., Imitrite and Acanthite compound formation with orthorhombic crystal structure, here it is evident that RBK which is intermediate compound of silver,[21] mercury, and sulfur whereas the final product RB is in silver sulfide form. Black sulfide of mercury and sulfur is used as media to prepare Bhasma has formed sulfide compound [Tables 1 and 2 and Graph 1].[22]

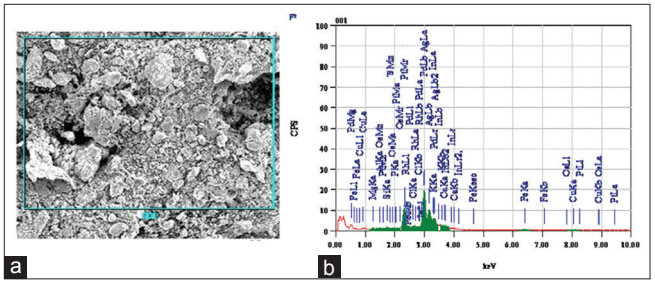
Bhasmas may be a compound of primary element used but considering the different variables in pharmaceutical processing such as media used for incineration, herbal juices used for lavigation, number of incineration cycles, and temperature gradient have a major role in transforming a metal into a medicament. As in previous studies, it was found in the oxide of silver,[23] wherein Haratala (arsenic trisulfide) and sulfur were used as media. However, in this study mercuric sulfide (HgS) and sulfur were used as media and RB was found to be in sulfide form. As detected in XRD final product in Ag_2S acanthite form in major but other constituents found in SEM-EDS, XRF which differentiates from pure laboratory chemical to therapeutic form.

SEM EDS results of RBK S 43.33%, Hg-31.74%, Ag 22.97%, K 0.57, Ca0.48, Na 0.47 in major,[21] and Mg 0.06 in minor level whereas RB processes Ag 75.63%, S 14.375, K 3.11%, Cu 1.68%, Ca 1.28%, Si 1.15%, Mg 1.09%, Fe 0.56%, Al 0.34%, and Rh 0.15% all in major elemental form Tables 3 and 4 and Graphs 2 and 3.

Further to revalidate the absence of Hg, final product of Bhasma was sent for XRF. Studies reported RB containing



Graph 1: (a and b) X-ray diffraction of *Kajjali* and Rajata Bhasma



Graph 2: (a and b) Scanning electron microscopy-EDX of *Kajjali*

Table 1: XRD report of RBK[21]

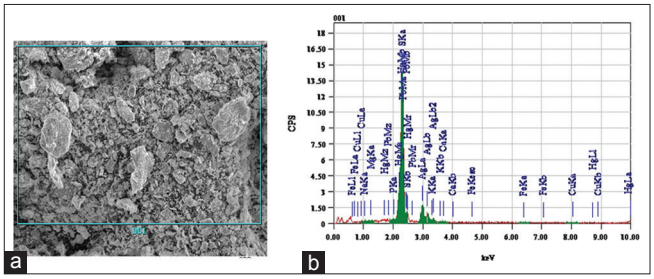
Compound name	Chemical formula	a/b/c (angstrom)	Crystal system	Bravais lattice	JCPDF
Imiterite	Ag_2HgS_2	11.28/8.98/6.78	Orthorhombic	Primitive (P)	00-023-1043
Calcium silicate	Ca_2SiO_4				00-039-0328
Pos. [2Th.]	d-spacing [Å]	FWHM [2Th.]	H	K	L
25.6491	3.47305	0.96	3	0	0
28.5954	3.12156	0.96	1	3	0
30.9881	2.88577	0.96	2	1	2
32.3198	2.76984	0.72	2	3	0
34.3969	2.60719	0.72	4	0	0
36.4492	2.46497	0.72	0	0	3
40.9777	2.20241	0.72	0	2	3
43.4733	2.08159	0.72	4	1	2
47.8396	1.90131	0.72	2	4	2
51.3243	1.78011	0.72	2	5	1
52.8598	1.73196	0.72	4	4	1
55.3714	1.6592	0.72	3	5	1
63.3421	1.46827	0.72	2	5	3

XRD: X-ray diffraction

Table 2: XRD report of RB

Compound name	Chemical formula	a/b/c (angstrom)	Crystal system	Bravais lattice	JCPDF
Acanthite	Ag_2S	19.96/4.90/3.44	Orthorhombic	Primitive (P)	00-009-0422
Pos. [2Th.]	d-spacing [Å]	FWHM [2Th.]	H	K	L
22.2723	3.99138	0.72	5	0	0
25.9591	3.43227	0.72	0	0	1
28.7925	3.10063	0.72	5	1	0
31.3427	2.85393	0.48	7	0	0
34.4124	2.60605	0.72	5	0	1
36.5925	2.45564	0.48	0	2	0
37.6694	2.38788	0.48	6	0	1
40.5839	2.22287	0.72	8	1	0
43.2769	2.09059	0.72	5	2	0
53.0648	1.72575	0.72	10	0	1
59.7536	1.54757	0.96	10	2	0
63.4926	1.46515	0.72	13	1	0

XRD: X-ray diffraction



Graph 3: (a and b) Scanning electron microscopy-EDX of Rajata Bhasma

Mg 0.0763%, Al 0.102%, Si 0.380%, P 0.252%, S 7.92%, K 3.31%, Ca 1.37%, Fe 0.612%, Cu 1.04%, Rh 0.32%, Ag 84.09%, and Os 0.0276% [Table 5] and [Graph 4]. Above analysis of RB indicating silver, sulfur, potassium, calcium and copper as major elements, and others in a minor amount. *A. vera* could be the source of K, Ca, Mg, P, Fe, and Cu used for Bhavana (levigation).[24] Concentration of silver and sulfur is very near to the theoretical stoichiometry of Ag2S. This finding is supportive of formation of silver sulfide compound detected in XRD. There is probability of multiple compound

formations like some part of the product may also be in oxide form however it was not traceable in XRD analysis.

Absence of Hg in final compound is evident in both XRF and SEM-EDX results. Bhasmas prepared out mercury as media is said to be superior.[25] Mercury may not only act as a catalyst to enhance the reaction but also imbibes therapeutic potential in the Bhasma.

Mean particle size of intermediate product was 6.70 µm ranging from 0 to 150 µm[21] and RB was 1.04 µm, 100% of the sample was found to be within 0-5 µm range [Table 6 and 7 and Graph 5]. Triturating with media and *A. vera* juice followed by incineration cycles has brought down the mean particle size along with the span value of RB. Particle size reduction is combined effect of trituration and heat. As trituration reduces the particle size and allows larger

Table 3: SEM-EDS report of RB before processing[21]

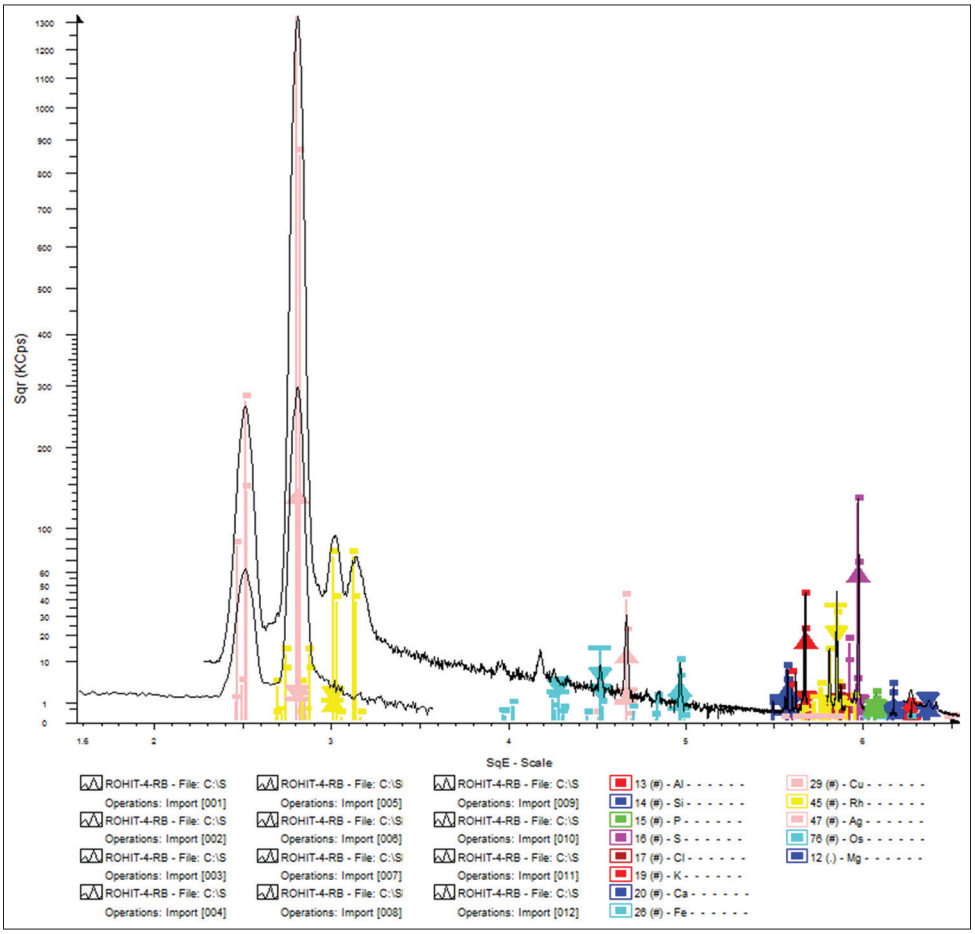
Element	(keV)	Mass %	Error %	At %
Na K	1.041	0.47	0.24	1.15
Mg K	1.253	0.06	0.15	0.69
S K	2.307	43.33	0.13	75.73
K K	3.312	0.57	0.26	0.81
Ca K	3.690	0.48	0.31	0.68
Ag L	2.983	22.97	0.60	11.93
Hg M	2.195	31.74	0.60	8.87
Total		100.00		100.00

SEM: Scanning electron microscopy

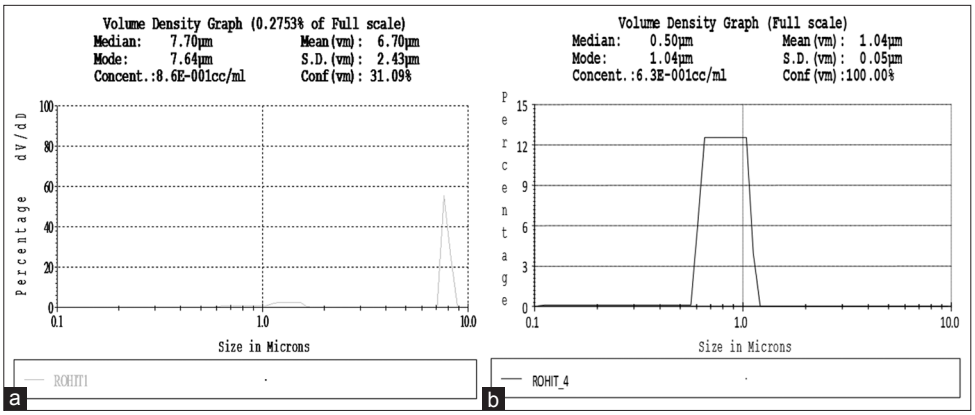
Table 4: SEM-EDS report of RB

Element	(keV)	Mass %	Error %	At %
Mg K	1.253	1.09	0.40	3.15
Al K	1.486	0.34	0.36	0.88
Si K	1.739	1.15	0.33	2.90
S K	2.307	14.37	0.33	31.64
Cl K	2.621	0.77	0.29	1.53
K K	3.312	3.11	0.47	5.62
Ca K	3.690	1.28	0.55	2.25
Fe K	6.398	0.44	0.89	0.56
Cu K	8.040	1.68	1.69	1.87
Rh L	2.212	0.15	0.89	0.10
Ag L	2.983	75.63	0.81	49.50
Total		100.00		100.00

SEM: Scanning electron microscopy



Graph 4: X-ray fluorescence of Rajata Bhasma



Graph 5: (a and b) Particle size of Rajata Bhasma

Table 5: XRF results of RB

Elements	Mg	Al	Si	P	S	Cl	K	Ca	Fe	Cu	Rh	Ag	Os
%	0.07	0.10	0.38	0.25	7.92	0.29	3.31	1.37	0.61	1.04	0.32	84.09	0.02

RB: Rajata Bhasma

Table 6: Particle size of RBK[21]

Samples	Particle ranges (%)	Mean particle size
RB before processing	0.0-5.0 µm (0.04) 5.0-10.0 µm (0.28) 80.0-85.0 µm (14.29) 125.0-130.0 µm (50.84) 145.0-150.0 µm (100.00)	6.70 µm

RB: Rajata Bhasma

Table 7: Particle size of RB

Samples	Particle ranges (%)	Mean particle size
RB	0.0-5.0 µm (100)	1.04 µm

RB: Rajata Bhasma

surface area to react when heated, thus making the metal into compound form.

CONCLUSION

RB prepared by Kajjali (black sulfide of mercury) as media was found in Ag2S (acanthite) with orthorhombic crystal structure with Ag in 75.63% and S in 14.375%. The particle size of the final product was within 1.04 µm. Study also showed there was absence of Hg and other heavy metals in the product.

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