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ORIGINAL RESEARCH ARTICLE PHYSICOCHEMICAL CHARECTERIZATION OF RAJATA BHASMA

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ABSTRACT

Rajata, a noble metal is used since long time in *Ayurveda* for its various therapeutic uses. In this study *Rajata Bhasma* was prepared classically and analysed through classical as well as modern analytical techniques. *Rajata* was subjected to *Samanya* (General Purification) and *Vishesha Shodhana* (Special method of Purification). *Shodhita Rajata Patra* (Thin sheets of purified silver) was made into small pieces, triturated with equal quantity of *Hingulottha parada* to obtain *Dhatu pishti*. *Shodhita Gandhaka* was added to it and *Kajjali* was prepared. *Kumari swarasa bhavana* was given to that *Kajjali* and later it was subjected for classical *Laghu Putapaka* . 20 *Laghu putas* were required to fulfil *Bhasma Siddi laxanas* which are *Nischandratva, Rekhapurna, Varitara* etc. The sample of *Rajata bhasma* was analysed using X-Ray Diffraction (XRD). The XRD peaks of *Rajata bhasma* were identified as silver sulphide (Ag_2S). Scanning Electron microscope with the Energy Dispersive X-ray (SEM-EDAX) instrument was also used for analysis, which showed Silver to be 74.16% while Sulphur was 13.34% along with other minerals. Particle size was found to be 54.16nm.

Keywords: *Rajata Bhasma*, XRD, SEM-EDAX, *Shodhana*, *Marana*

INTRODUCTION

Bhasmas are unique preparation of *Ayurveda*, used in the treatment of various diseases. They have great therapeutic value because they get absorbed easily in the body even in very small dosage. However if these *Bhasmas* are not well prepared they can be toxic to human body. Therefore, *Bhasma Pariksha* is given in *Ayurveda* to confirm the well prepared metallic *Bhasma*, but in this era we need to analyze the *Bhasmas* on modern parameters too to make it acceptable globally. XRD & SEM-EDAX analysis are the important technique by which compounds of material, composition of various material, particle size and free metals etc can be detected.

Rajata Bhsama is used for the treatment of *Madumeha* (Diabetes), *Apasmara* (Convulsions), *Pandu* (Anaemia), *Shwasa* (Asthma) etc. It is a potent *Rasayana* (Rejuvenative) drug as well. Hence *Rajata bhasma* was used for the present study.

Materials and methods

Prepararion of *Rajata Bhasma*:

***Shodhana* of *Rajata*[1]:**

The purification of *Rajata* was carried out in two stages. First stage been the *Samanya shodhana* were in *Rajata,* was made into *Patra* (thin sheets) form and subjected to 7 times *Nirvapa* (heating till red hot and quenching in liquid media) in *Tila Taila*, *Takra, Gomutra, Kanji* and *Kulattha Kwatha* subsequently. After every *Nirvapa*, the liquid medium was changed.

Vishesha Shodhana[2]**:** *Samanya Shodhita Rajata* was again heated till red hot and quenched in *Nimbu Swarasa* for seven times. Juice was changed after every dipping. Finally *Shuddha Rajata* was collected carefully.

***Marana*[3]:**

***Pishti* formation**: S*hodhita Rajata* (125gm) was converted into tiny piece and triturated along with equal quantity of *Hingulottha Parada*[4] (125gm) for 20 hrs. It was converted to a lustrous steel gray colour coarse powder instead of *Pishti*.

The above obtained coarse powder was then added with *Shuddha Gandhaka*[5] (equal quantity to *Rajata*) and triturated for about 42 hrs, to obtain *Kajjali*.

***Putapaka*[5]:** The above *Kajjali* was triturated with *Kumari Swarasa* (Aloevera) till a homogeneous paste was formed which took 6 hours time. *Chakrikas* (Small pellets of uniform size and thickness) were prepared and dried in sunlight. These dried pellets were kept inside a *Sharava* (shallow earthen disc) and another *Sharava* was inverted over it. The joint between the two discs was sealed with a *Kapad mitti* (a ribbon of fine cloth uniformly smeared with fuller's earth) and dried in sunlight. This procedure was repeated seven times to assure proper *Sandhi bandhana*. The properly sealed and dried *Samputa* was subjected to classical *Laghu puta*[6] (with 2 kg cow dung cake).

After cooling, the *Chakrikas* were triturated adding *Kajjali* (In half the proportion to Rajata i.e., 62gms) and *Kumari swarasa*. The homogenous mixture was again subjected to *Laghu Puta*. The same procedure was repeated for 3rd *Puta* also. From 4th *Puta* onwards *Kajjali* was not added and *Bhavana* of *Kumari Swarasa* alone was used. 20 *Laghu putas* were required to attain *Bhasma siddhi Laxanas*. In initial 10 *Putas* heat was gradually increased and it was gradually decreased in subsequent 10 *Putas*. Approximate temperature for the preparation of *Rajata Bhasma* was 500 – 550 ^{O}C. The *Bhasma* obtained from the above process was taken for analysis.

Analytical study of Rajata Bhasma:

Classical parameters opted: *Varna, Sparsha, Gandha, Varitaratva, Rekhhapoornatva, Unama, Gatararasatva, Nishandratva, Apunrbhava, Niruttha* Test.

Physico-chemical Parameters Opted: pH value, Total Ash, Acid insoluble ash, Water soluble ash, Loss on drying, Estimation of silver, Mercury, Sulphur, Iron content (Manual Method) [7].

Analysis using modern parameters

The *Rajata bhasma* (R.B), *Shodhita rajata* (S.R) as well as the raw material (*Ashodita rajata*) were analysed using following techniques to know the changes which take place in each process.

1 .X-ray diffraction

2. Scanning Electron Microscope (SEM) with the Energy dispersive x-ray.

RESULTS:

Table 1: Showing classical parameters for Analysis of R.B.

Sl No	**Parameter**	***Rajata Bhasma***
1	*Varna*	*Krishna*
2	*Sparsha*	Smooth, fine (*Mrudu*)
3	*Gandha*	*Nirghanda*

4	*Varitaratva*	Floats on surface of water
5	*Rekhapoornatva*	Fills the space between finger lines
6	*Unnama*	Grains of rice float on the *Bhasma* floating on water
7	*Gatarasatva*	Tasteless
8	*Nischandrata*	No lustrous particles observed
9	*Niruttha*	Weight of silver does not increased
10	*Apunarbhava*	*Bhasma* does not return to original metal form

Physical and Chemical parameters of *Rajata Bhasma* has also been checked by manual method.

Table 2: Showing result of Physical Tests of R.B

Contents	**Rajata Bhasma**
pH value	5.43 ± 0.10
Total ash	99.50
Acid Insoluble ash	11.50
Water Soluble ash	Nil
Loss on Drying	0.00%

Table 3: Showing result of Chemical tests of R.B

Contents	**Rajata Bhasma**	**Shodhita Rajata**	**Ashodhita Rajata**
Silver	70.72%	83.30%	96.30%
Silver as sulfide	76.32%		
Silver as oxide	1.23%		
Free Mercury	Nil		
Mercurous	Nil		
Mercuric	Nil		
Sulphur	10.34%		
Free sulphur	Nil		
Sulphide	9.25%		
Sulphate	1.09%		
Iron	2.00%		
Ferrous	1.67%		
Ferric	0.33%		

In modern advanced parameters X-ray diffraction (XRD) was done to crackdown the structure and chemical composition of the samples. (**Fig1, 2, 3 & Table 4**)

X-ray diffraction study[8]

X-ray diffraction studies were performed in the Indian Institute of Science (IISc), Bangalore. It was carried out by using JEOL JDX 8p X-ray diffractometer. The strongest peak identified in the raw material was Ag and in the *Shodita Rajata* it was identified as Silver oxide (Ag_2O), while that in the final product was Silver sulphide (Ag_2S).

Table 4: X-ray diffraction of the *Ashodita Rajata* (A.R), *Shodita Rajata* (S.R) and *Rajata Bhasma* (R.B)

Parameters	**A.R**		**S.R**		**R.B**	
	Peak	Rel. Int%	Peak	Rel.Int%	Peak	Rel.Int%
XRD identified peaks with Rel. Int%	38.261, 44.375, 64.487, 77.441	38.20 20.14 100.00 8.82	38.2470, 64.5315, 77.5106	100.00 48.48 50.71	26.0086 31.6274 33.7759 34.5449 36.9862 37.8416 40.8753 43.5917 44.3622 46.3100	9.65 27.33 11.28 29.38 22.87 32.41 8.73 12.26 13.59 9.39
Name	**Silver**		**Silver oxide**		**Acanthite**	
Composition	**Ag**		**Ag_2O**		**Ag_2S**	
Crystal structure system	**Cubic (face centered)**		**Cubic**		**Orthorhombic**	

Figure 1: X-ray diffraction of Ashodita Rajata

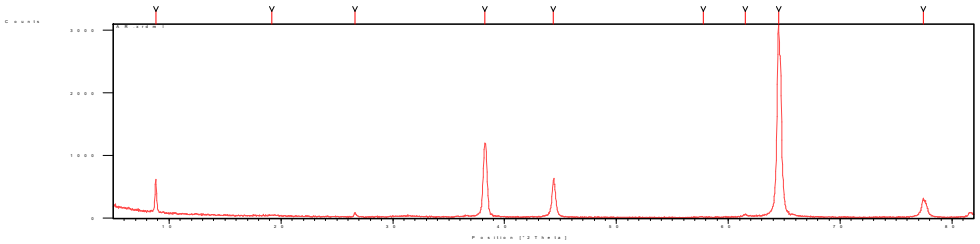


Figure 2: X-ray diffraction of Shodita Rajata

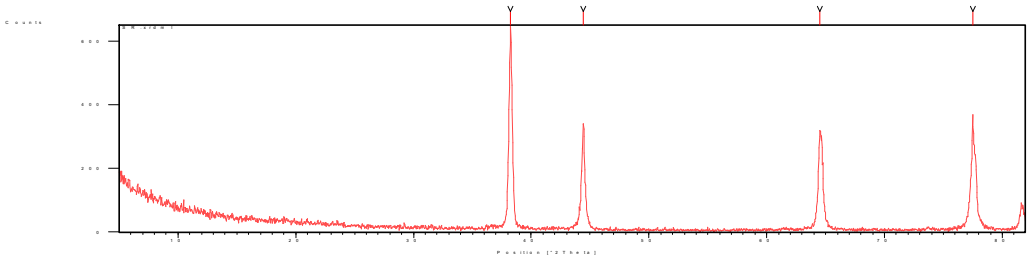
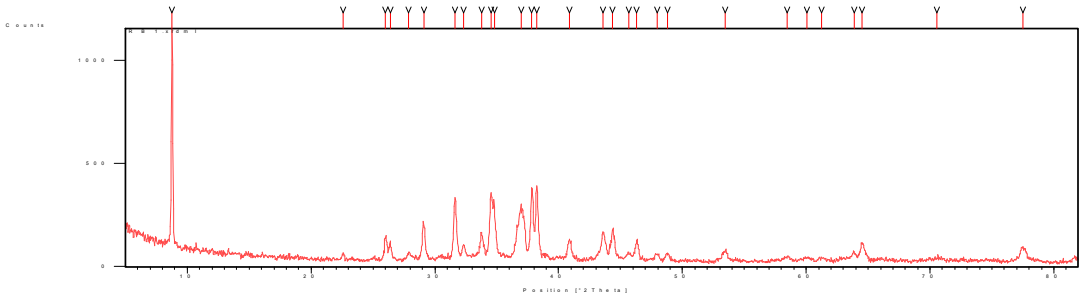


Figure 3: X-ray diffraction of Rajata Bhasma



Study using Scanning Electron microscope with the Energy Dispersive X-ray study (SEM-EDAX) [9]**:**

Quantitative elemental analysis of *Ashodhita Rajata, Shodhita Rajata* and *Rajata Bhasma* & particle size analysis is carried out with the help of Scanning Electron Microscope with the Energy dispersive x-ray (SEM-EDAX) analysis at Dept. Of Nano Science and Engineering, Indian institute of science, Bangalore

Table 5: Showing result of EDX of A.R, S.R and R.B

	Concentration in%		
Elements	**A.R**	**S.R**	**R.B**
C	0.00	0.00	1.30
O	**0.00**	**13.01**	**2.64**
Na	0.83	0.41	1.12
Mg	0.00	3.03	0.77
Si	0.00	1.21	1.27
S	**0.00**	**0.00**	**13.34**
Cl	0.00	0.00	0.21
K	0.00	0.21	4.09
Ca	0.00	0.00	2.19
Cu	0.00	1.02	1.04
Ag	**99.17**	**81.11**	**74.67**
Hg	0.00	0.00	0.00
Pb	0.00	0.00	0.00

Figure 4: The SEM-EDX spectra images of *Ashodita Rajata*

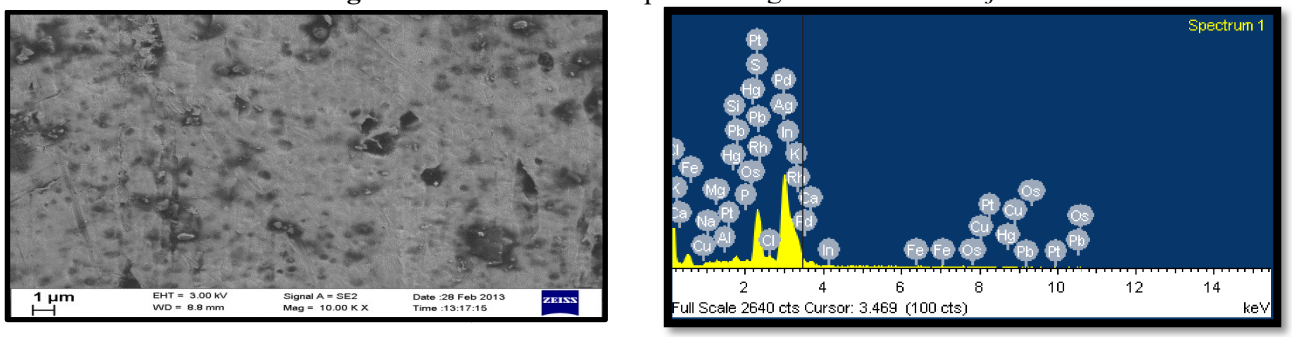


Figure 5: The SEM-EDX spectra images of *Shodita Rajata*

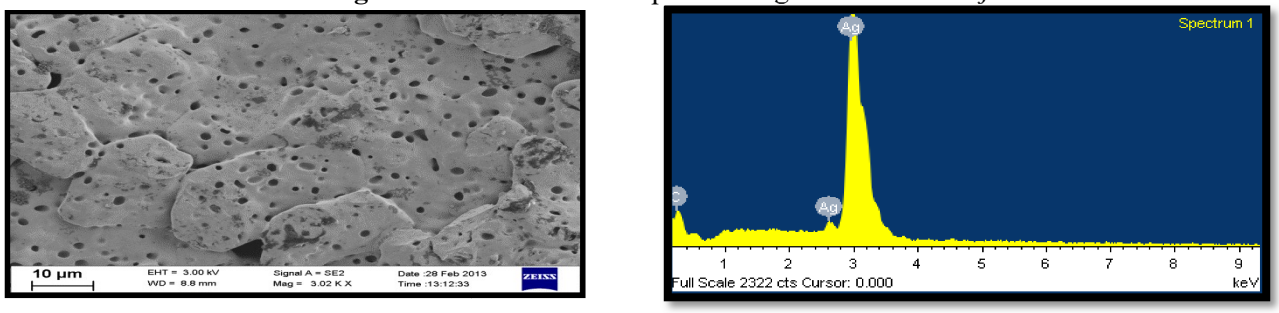
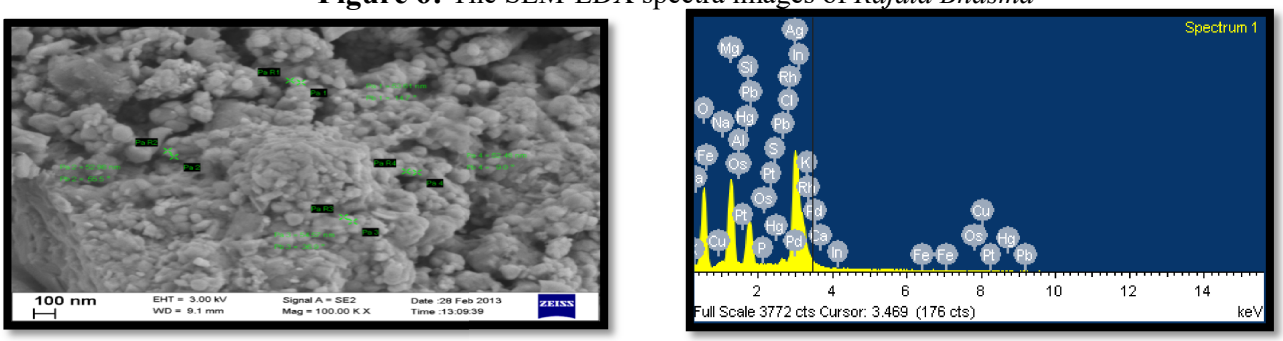


Figure 6: The SEM-EDX spectra images of *Rajata Bhasma*



The detected elements of *Ashodhita Rajata* revealed that Silver (99.17%) is the major element and Sodium (0.83%) is the minor element. The analysis of *Shodhita Rajata* revealed that Silver, Oxygen, Magnesium, Silica and Copper are the major elements whereas Sodium and Potassium are the minor elements. While the analysis of *Rajata Bhasma* revealed that Silver, Sulphur, Potassium, Calcium, Oxygen, Carbon, Copper and Silica are the major elements and Magnesium is the minor element.

The particle size analysis is carried out with the help of SEM and images show reduced particle size of Rajata *Bhasma* in Nanometre range i.e. **54.16 nm**.

Table 6: Showing Particle Size of *Rajata Bhasma*

Name of the sample	Particle size range		Mean particle size
Rajata Bhasma	14.7^{O}	56.61 nm	54.16 nm
	56.5^{O}	52.98 nm	
	36.9^{O}	54.57 nm	
	90^{O}	52.48 nm	

DISCUSSON

In *Rasashastra* literature *Bhasmikarana* is an important pharmaceutical procedure which converts the metals/minerals into desired compound form, suitable for internal use i.e. into a very fine, absorbable state which is therapeutically most effective and least or non-toxic form.

The *Rajata Bhasma* passed the *Rekhapurnata*, *Varitara and Unam* tests indicating the reduced particle size, more surface area and *Laghuta* of bhasma.

The samples of *Bhasma* passed the *Apunarbhava and Niruttha* test indicating stable form of *Bhasma. Nischandratva* of *Bhasma* was also observed.

Rajata bhasma is having pH of 5.37± 0.10**.** The value indicates the weak acidic nature of the drug. Total Ash value of R.B was 99.50%. And it is accurate according to the pharmacopeia standards of *Bhasma*. High ash value in R.B shows the presence of very high inorganic content.

In the Loss on drying at 110^{0}C study R.B possesses 0.00% loss on drying. Hence it can be stated that R.B have no moisture content and very rare chance of bacterial and fungal growth.

Estimation of forms of Mercury such as Mercurous mercury and Mercuric mercury was nil in *Rajata bhasma*, so it indicates proper formation of *Bhasma* as per pharmacopeia standards and it shows, mercury might have a specific role to facilitate the metal for the formation of *Bhasma*.

Estimation of Sulphur was done (Table 3) showing 10.35 % of sulphur. There was absence of free sulphur in the sample which is considered safe. The sulphide form was significantly more in comparison to sulphate form.

The Estimation of silver compounds was done (Table 3). The silver content was reduced to 83.30% after *Shodhana*. During *Nirvapa* silver was converted to Silver oxide. This conversion of Silver into oxide form leads to decrease in silver content after *Shodhana* procedure. And during Marana silver percentage has reduced further because of partial conversion of silver into sulphide form. By observing the results it can be concluded that R.B is the combination of sulphides (in major) and the oxides (in minor) of silver.

According to the need of time, characterization of *Bhasma* using scientific techniques is necessary to determine the effect of the process and to judge its safety and efficacy. *Rajata Bhasma* was prepared and studied with this objective. XRD study of the raw material showed a sharp peak, indicating its crystalline nature; whereas for final product intensity of peaks was less, indicating the loss of crystalline nature, which justifies *Nischandratva*, a classical procedure to know *Bhasma siddhi laxana*. XRD study of *Shodita Rajata* showed Ag_2O peak, indicating that some part of silver was transformed to Silver oxide. This substantiates that the metal is transformed to compounds in *Shodhana* steps too. Here from S.R to R.B change in chemical nature and crystal structure is seen which might be due to the effect of heat and specific procedure adopted in *Bhasmikarana*, resulting in the Oxidation and reduction reaction and resulting in compound Ag_2S which is in amorphous state.

The SEM-EDX study of *Ashodita Rajata* showed the percentage of Silver is 99.17%, which indicate the high purity of raw material. The analysis of S.R revealed that Silver, Oxygen, Magnesium, Silica, Copper are the major elements on the other hand Sodium and Potassium were minor elements. It's because of repeated *Nirvapa*, porosity develops in the *Rajata* foil, bonding between silver atoms becomes loose and some bonds are seen to break up. During the *Nirvapa*, inorganic and organic compounds present in liquids come in contact with hot foils of silver and dissociate due to high temperature (700^o C) and may react with the silver atoms. Thus few elements like Magnesium, Silica, Copper, Sodium, Oxygen and Potassium infuse with the silver atoms. Analysis of *Rajata Bhasma* indicated Silver, Sulphur, Potassium, Calcium and Copper elements in Major. Here increase in percentage of Calcium and Potassium may be due to use of *Kumari swarasa* which is containing these elements in organic form.

Presence of silica in *Rajata Bhasma* may be due to the use of earthen casseroles, which may have a reaction with oxygen. Presence of Sodium, Potassium, and Phosphorus may be due to use of *Nimbu* swarasa which is containing these elements in organic form. Oxygen and Carbon these elements were also present, due to oxidation process.

The SEM images show reduced particle size of *Rajata Bhasma* in Nanometre range i.e. **54.16 nm**. It is this significant reduction of size that allows the phenomenon of *Rekhapurnata, Varitara* and *Sukshmata* of the *Rajata Bhasma.* Particle size is one of the factors which will affect dissolution and absorption of drug. Particle size and surface area are inversely proportional to each other, as particle size decreases surface area increases. This leads to increase in dissolution of drug and rapid absorption. Again the cluster of particles is regular and uniform in the final *Bhasma* in comparison to the raw material. Here the consequent heat treatment in the form of *Puta* and wet grinding in the form of *Bhavana* may be responsible for reduction in particle size. The particle size can be characterized as the desired specification of *Bhasma.*

CONCLUSON

Based on the physical tests R.B. is weak acidic in nature. Total Ash, acid insoluble ash, water soluble ash and loss on drying are within the acceptable limits reflecting its genuinity. Chemical tests denote that the drug does not contain free mercury and sulphur which proves its safety. A major percentage of Silver is in Silver Sulphide form and sulphur in sulphide form. As per XRD report, *Ashodita Rajata*

is in the form of Ag with Cubic crystal structure, the compound identified as Silver. *Shodita Rajata* is in the form of Silver oxide with Cubic crystal structure and *Rajata Bhasma* in the form of Acanthite with Orthorhombic crystal structure. Mean Particle size of R.B is **54.16 nm**. This reduction in the particle size into nanometre range, owing to the rigorous pharmaceutical procedures will aid in the precise drug delivery and increasing the bio availability of the drug.

The structural and chemical transformation of metal into metallic compounds (*Bhasma*) which are bio absorbable and assimiable are the main objectives of *Marana*. To avoid any toxicity and adverse effects of *Bhasma*, the complete transformation of base metal into *Bhasma* form is prime requisite. To check whether the *Bhasma* is properly formed or not, *Rasashastra* texts have laid down certain *Bhasma parikshas* (tests). These *Bhasma Parikshas* are qualitative in nature and are enough to prove properly formed *Bhasma*. But in this scientific era analysis of *Bhasma* with modern analytical parameters like XRD, SEM-EDAX is very useful for it reveals about the characterization of the *Bhasma* like particle size, components of *Bhasma* etc. Results indicate that the advanced modern parameters are really the necessity to understand the *Bhasma* scientifically.

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