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Research Article

PHYSICO-CHEMICAL ANALYSIS OF *SHADGUNA KAJJALI* AND *SHADGUNA BALIJARITA RASASINDURA*)

Sorab Gaind[1]*, Pradeep Agnihotri[2]

*[1]Lecturer, Department of Rasa Shastra, Guru Nanak Ayurvedic Medical College and Hospital, Sri Muktsar Sahib, Punjab, India.

[2]Professor, Department of Rasa Shastra, Ayurveda Mahavidyalya, Hubli, Karnataka, India.

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ABSTRACT

Analytical study of any drug is essential to standardize it. Analytical study is carried out to check drug quality. For this purpose some analytical tests are performed and their results are compared with standard parameters. The drug fulfilling these criteria can be taken as standard drug and can be used for therapeutic purpose. Percentage of total mercury in *Shadguna Kajjali* and *Shadguna balijarita rasasindur* was 40.03%, and 84.17%. Total Sulphur in *Shadguna Kajjali* and *Shadguna balijarita rasasindur* was 43.07% and 11.16%. Percentage of free sulfur in *Shadguna Kajjali* and *Shadguna balijarita rasasindur* was 22.11%. XRD of *Shadguna balijarita Rasasindur* were identified as Cinnabar (Hgs) with Hexagonal Crystal Structure having primitive lattice. In this paper Physico-Chemical Analysis of *Shadguna Kajjali* and *Shadguna balijarita rasasindura* is done.

INTRODUCTION

Ayurvedic Pharmacopoeia has prescribed several parameters for evaluating quality of single Ayurvedic herbal drugs. Parameters prescribed are; loss on drying, ash value, water soluble extracts, qualitative and quantitative analysis, etc. To obtain product of uniform quality, standardization is essential especially for *Rasa* preparations. Hence the raw materials, intermediate products and finished products were subjected to chemical analysis.

Though Ayurveda is having its unique analytical approach towards drugs, in present era there is a necessity of modern analytical techniques. For analysis and standardization of *Rasaoushadhis*, knowledge of analytical chemistry is very much essential. So the analytical methods adopted in the present study and their applications are reviewed.

Analysis means a detailed examination of substance in order to interpret or explain it. Chemistry is concerned with the properties and interactions of the substances of which matter is composed. Analytical chemistry is a tool to gain information about the qualitative and quantitative composition of substances and chemical species, i.e. to find out what a substance is composed of and exactly how much.[1]

AIMS AND OBJECTIVES

1. To prepare *Shadguna balijarita Rasasindura* as per classical reference.
2. Physico-chemical analysis of *Shadguna balijarita Rasasindura*.

Table 1: Showing Classical Parameters for analysis of *Samaguna & Shadguna Rasa Sindura*

Test	Observation
Varna	*Sindur*
Sparsh	*Slakshnamrdu*
Gandha	Slight sulphur smell
Rekhapurnatva	When fine powder of *Rasa Sindur* was rubbed between the thumb and index finger it entered the furrows of the fingers

Varitaratva	When finely powdered *Rasa Sindur* was carefully sprinkled into a test tube containing water, *Kajjali* was floating over the water.
Nischandratva (Lustreless)	No shining particles were observed.

Modern Parameters

Table 2: Physical Tests

Organoleptic Characters	***Shadguna Kajjali***	***Shadgunabalijarita Rasa sindura***
Colour	Greyish Black	Reddish brown
Odour	Tinge sulphur	Odourless
Touch	Fine powder	Fine powder
Taste	Tasteless	Tasteless

Determination of pH Value[2]

Materials

Glass electrode, pH meter, Buffer tablet

(PH - 4) Acid - 0.05H Potassium hydrogenphthalate,

(PH – 8) Alkali - 0.05H Sodium tetraborate.

Beakers

Shadgunakajjali and *Shadguna Rasasindura* each-1gm.

Method: Operate the pH meter and electrode system according to the manual instructions. When measuring an alkaline solution, standardizing the meter and electrodes with 0.05H sodium borate is done. At the end of a set of measurements, take the solution used to standardize the meter and electrodes. This reading should not differ more than 0.02 from the original value at which the apparatus was standardized. Now in 5ml of water 1gm of sample was put and pH is determined for the solution.

Determination of Ash Value[3,4]

Materials: Silica crucible, Electronic weighing machine, Electric furnace.

Shadguna kajalli, *Shadguna Rasasindura*– 2 gm

Method:

Two grams of accurately weighed sample was taken and transferred to the cleaned, dried and weighed. Silica crucible and was subjected to ignition using electric furnace at 450°C for an hour. Silica crucible was taken out from the furnace and was allowed to cool and weighed. After cooling, weight of the ash was obtained and the ash value of sample was calculated.

Determination of Acid Insoluble Ash[5,6]

Material: Silica crucible, Burner, Whatman's filter Paper, Electronic weighing machine, DilHCl - 25ml.

Conical flask

Ash of *Shadguna kajalli, Shadguna Rasasindura*

Method: 2gm of sample is dissolved with 25ml dilute hydrochloric acid for 5 min, then filtered through whatman's paper and was washed with water. The residue was taken in a crucible, dried and ignited, allowed to cool and weighed.

Determination of Water Soluble ash[7]

Material: Burner, Whatman's filter Paper, Electronic weighing machine, Water

Ash of *Shadguna kajjali, Shadguna Rasa sindura*

Method: Boil the ash for 5 minutes with 25ml of water. Collect the insoluble matter in an ash less filter paper and washed with hot water and is ignited for 15 minutes at a temperature not exceeding 450°C. Subtract the weight of the insoluble matter from the weight of the ash. The difference in the weight represents the water soluble ash. Calculate the percentage of water soluble ash with reference to the air dried drug.

Determination of Loss on Drying at 110°C[8]

Materials: Silica crucible, Electronic weighing machine, Electronic air oven

Shadguna kajjali, Shadguna Rasasindura each 1gm.

Method: One gram of sample was taken in a Silica crucible and accurately weighed, heated on electric air oven upto 110°C for 3 hrs. Again weighed the difference and weight was calculated.

Table 3: Physico-Chemical Analysis

	Shadguna Kajjali	***Shadguna balijarita Rasasindura***
pH value	8	7
Ash Value	0.13%	0.04%
Acid Insoluble Ash	0.09%	0.03%
Water Soluble Ash	0.02%	0.05%
Loss on Drying AT 110°C	0.23%	0.04%

Chemical tests

Estimation of Total Mercury by Volhard[9]

Method: Reagents

1. Concentrated Sulfuric acid
2. Potassium permanganate
3. Oxalic acid
4. Ferrous Sulphate Solution
5. Ferric ammonium Sulphate Indicator
6. Potassium thiocyanate Solution

Sample Preparation

Transfer known quantity of samples to Kjeldal flask fitted with short stemmed funnel add 5ml of concentrated Sulfuric acid and is mixed. Add 0.5 to 1gm of potassium permanganate was added in small portions with vigorous shaking. Rinse down with 5ml of conc. Sulphuric acid. Shake the flask for 30 minutes. Then heat is increased gradually to boiling and removed from heat without cooling. Small portion of oxalic acid was added until the manganese dioxide has been reduced and dissolved, cooled and diluted to 100ml.

Method: A known quantity of solution is taken in a conical flask. Any Mercurous mercury or Nitrogen oxides was oxidised by adding 0.1M potassium permanganate solution drop wise by stirring until the pink color persists for 5 minutes. Remove excess of permanganate by adding just enough 0.1M Ferrous Sulfate Solution. 1.5ml of ferric ammonium sulfate indicator was added, cooled to 15^0C and titrated with potassium thiocyanate solution.

Hg % = (V) (A) 100

W (1000)

V= Volume of the thiocynate solution

A= Mercury equivalent of thiocynate

W= Sample weight contained in aliquote

Estimation of Free Mercury by Ion Selective Eletrode[10]

Materials: Ion selective electrode, Beaker

Shadgunakajjali, Shadguna Rasasindura each 1gm.

Method: A known quantity of sample in a beaker was taken. 100ml of water added and filtered. The filtrate was collected. Aliquot of the sample is taken and analysed the free mercury by ion selective electrode method.

Estimation of Sulphur By Eschka Method (Gravimetrically) [11]

Eschka mixture and other reagents

Electronic weighing machine

Crucible

Whatman filter paper

Shadguna kajjali, Samaguna and *Shadguna Rasasindura* each 1gm.

Method: 1gm of sample is ground to pass 80 mesh sieve and 3gm of Eschka mixture (2 part of calcined magnesium oxide and 1 part of anhydrous sodium carbonate) is added. It is mixed in a crucible and covered with another 2 grams of Eschka mixture. the content ignited till all the carbon is burnt and the crucible was cooled. 10% of barium chloride solution was added by constant stirring to precipitate all the sulphates. The solution was filtered with Whatman filter paper and the precipitate was collected and weighed it as Barium Sulphate.

Estimation of Free Sulphur

To find free Sulphur, further calculations were done on the basis of percentage of total Sulphur.

Table 4: Elemental Analysis

Contents	***Shadguna Kajjali***	***Shadguna balijarita Rasasindura***
Total Mercury	40.03%	84.17%
Free Mercury	Traces	Nil
Total Sulphur	43.07%	11.16%
Free Sulphur	22.11%	Traces

X-Ray Diffraction Study

Materials

Brukar's D-8 Advance X-ray diffractometer and is equipped with Cu K-alpha (Lambda-1.5 406) radiation and graphite monochromator operated at 40KV/30mA.

Samaguna & Shadguna Rasa Sindur each 1 gm.

Method: Sample was well grounded to 200mesh and air dried. The X-ray diffractometer scans were made on randomly oriented samples form 3-650 2-theta (d=29.42 to 1.43angstorm) with a step size of 0.020 and one second time per step.

The 2-theta value and intensity of the peak (counts) are represented on X and Y-axis respectively. Higher the value of counts represents higher the crystallanity of the phase.

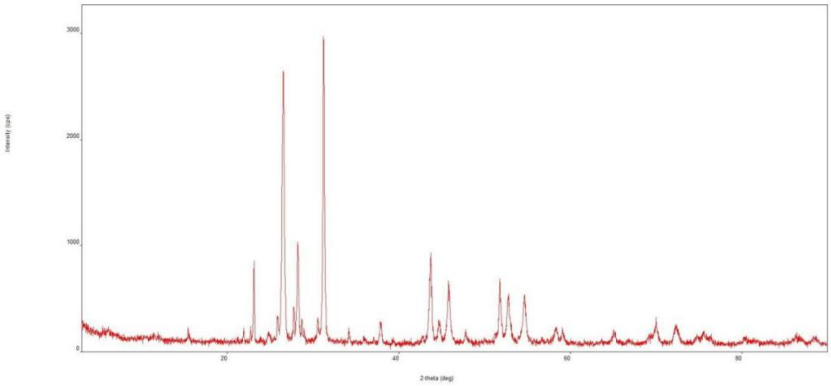
For identification of each phase, minimum 6strong peaks were chosen and compared with standard X-ray Powder Diffraction file (XPDF).

Table 5: Showing XRD of *Shadgunabalijarita Rasasindur*

Identified			Standard	
Peak no.	**Angle 2 θ**	**Intensity**	**Relative Intensity%**	**Relative Intensity %**
4	23.08	343.00	92	100
6	26.500	1059.00	32	30
8	28.200	415.00	100	98
11	31.200	1189.00	21	25
14	45.800	267.00	20	20
16	45.880	231.00	19	25

Name of Standard: Cinnabar (Hgs)
Crystal Structure: Hexagonal
Lattice: Primitive

1. Around 28 peaks were identified in *Shadguna balijarita Rasasindura* sample at different angels (2θ) from 20.64 to 88.5.
2. 6 strong peaks were chosen as strong with their relative intensity and compared to standard X–ray powder diffraction file (XPDF).
3. 11th peak with relative intensity of 21% was considered as significant at 31.200
4. The intensity % of Cinnabar (100, 30, 95) was approximately matching with the intensity % of sample (92, 32, 100) respectively.



Graph Showing XRD of *Shadgunabalijarita Rasasindur*

EDAX/EDS STUDY

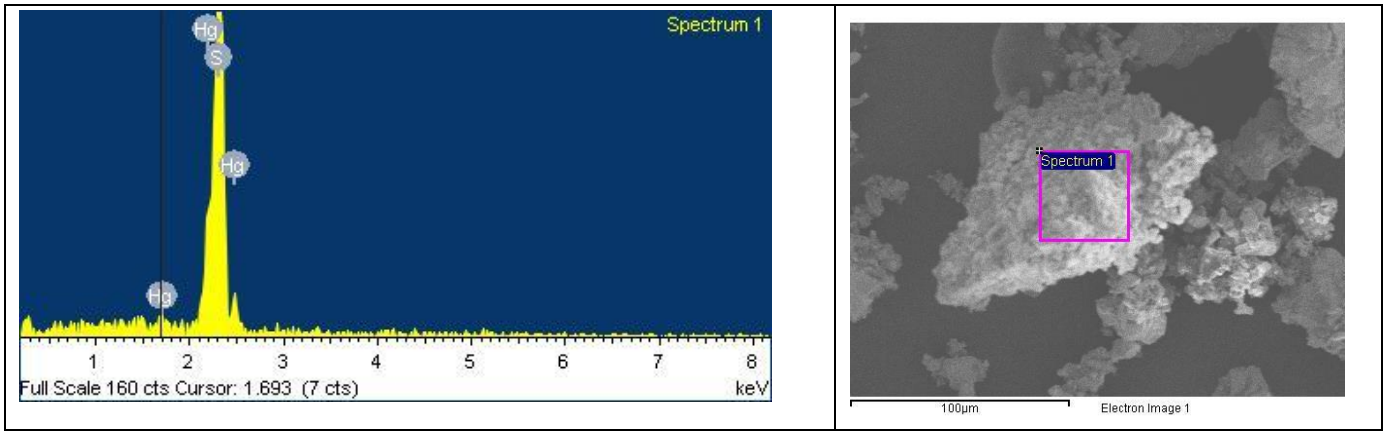
EDAX refers to energy dispersion X-ray spectroscopy and is employed here as an analysis tool to determine the elemental composition of *Shadguna Rasasindura*. It works by analyzing the spectrum of emitted x-rays from a sample as a beam of high energy electron is incident upon it. Here EDAX worked in association with SEM. EDAX is a semi quantitative method of estimation as it is not taking account of the entire sample, but only the area of focus. But, being a non destructive method, it remains a very valuable tool as the elements that are destroyed by the destructive techniques like ICP; AAS can be estimated by this method. In the present study after thoroughly mixing the sample, SEM images captured randomly at 6 different areas were connected to the EDAX spectroscopy to generate elemental composition in both mass% and atomic%.

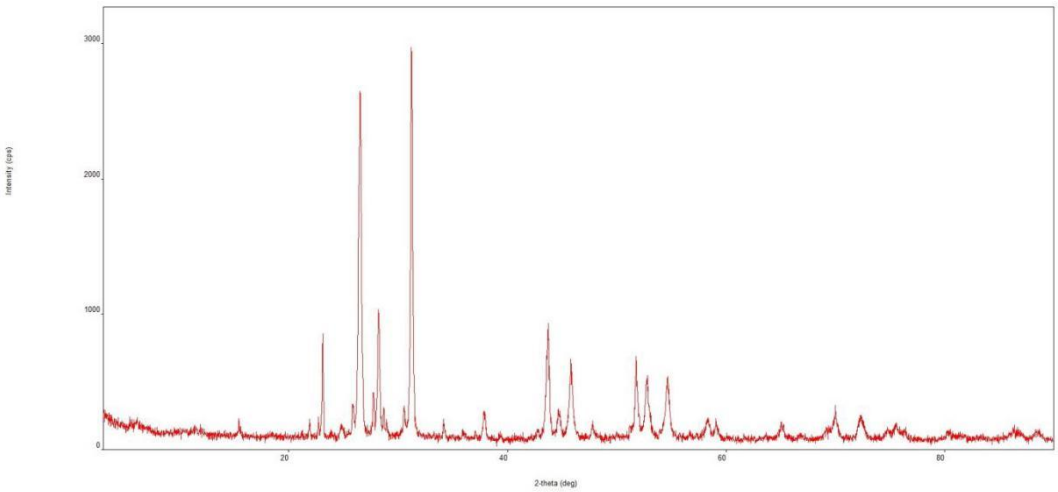
Element identified are Hg, S

Shadguna Rasasindura

Spectrum processing:
Peak possibly omitted: 0.146 keV
Processing option: All elements analyzed (Normalised)
Number of iterations= 2
Standard:
FeS2 1-Jun-1999 12:00 AM
Hg HgTe 1-Jun-1999 12:00 AM
S FeS2 1-Jun-1999 12:00 AM
Hg HgTe 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
S K	54.31	88.15
Hg M	45.69	11.85
Totals	100.00	100.00





EDAX Study of *Shadguna Balijarita Rasasindura*

SEM Study

SEM and EDS reports

The SEM (scanning electron microscopy) image formed is the result of the intensity of the secondary electron emission from the sample at each x, y data point during the rastering of the electron beam across the surface. From the images morphology and particle size can be calculated.

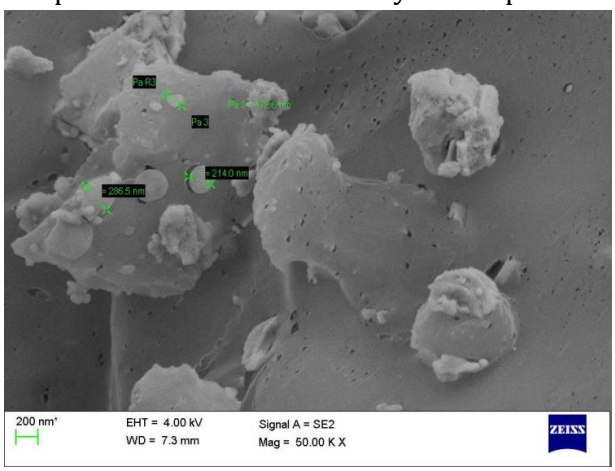
Along with the secondary electron emission, which is used to form a morphological image of the surface this electron scanning provide elemental analysis by the attachment of an Energy-Dispersive Spectrometer (EDS). X-ray emission results from inelastic scattering between the beam electrons and the electrons of the sample atoms. This interaction results in the ejection of an inner shell electron from the atom creating a vacancy that is filled by an outer shell electron. This jump from an outer to inner shell results in a change in energy that produces either a X-ray or Auger electron. The emitted x-ray has energy equal to this change. The x-rays are then detected by either a lithium-drifted silicon detector for an EDS system and can be used to identify the chemical composition.

Here the SEM pictures with EDS were recorded using Ultra 55 Field emission scanning electron microscope with EDS (Karl Zeiss).

Individual description for all samples best picture is of 200nm (one has the particle size included).

Shadguna Balijarita Rasasindura

The surface is ultra smooth and porous as shown by SEM. Some small particles of nanometer size can be seen on the surface. The EDS shows the chemical composition consists of mercury and sulphur.



Comparative Analytical Study

Table 6: Showing Comparative Results of Physical Tests

Contents	***Shadguna kajjali***	***Shadguna balijarita Rasasindura***
Color	Greyish black	Reddish brown
Odour	Odourless	Odourless
Touch	Smooth	Fine powder
Taste	Tasteless	Tasteless
Ph value	8	7
Ash value	0.13%	0.04%
Water soluble ash	0.02%	0.05%
Loss on drying	0.23%	0.04%

Table 8: Showing comparative XRD results of *Shadguna balijarita Rasasindur*

Angle 2θ	**Intensity count/sec**	**Intensity**
23.08	343.00	92
26.50	1059.00	32
28.20	415.00	100
31.200	1189.00	21
45.80	267.00	20
45.880	231.00	19

Table 9: Showing Comparative Results of Zeta Potential

Sample	**Zeta Potential (mV)**	**Stability behavior of sample**
Shadguna Rasasindura	-22.23	Incipient

DISCUSSION

Physical appearance of both the *Kajjali* and *Rasasindhooraa* were same, as ingredients and method of preparation were same.

***Kajjali*:** The obtained *Kajjali* was black fine powder and possessed *Slakshnatva* and *Sukshmatva* which indicates the fineness of *Kajjali* attained by doing pressurized, uniform and continuous *Mardana*. *Rekhapurnatva* denote the fineness in particle size i.e., size has been reduced so as to enhance bio-availability. *Nishchandratva* denote the absence of free mercury state in *Kajjali*.

***Rasa Sindura*:** *Shadguna balijarita Rasasindura* was obtained as brownish red shiny conical blocks. The color of finely powdered *Sindura* was reddish brown. *Nishchandratva* indicate absence of mercury in elemental form. *Varitaratva* confirmed the fineness of the product.

Discussion on pH

pH of *Shadguna Kajjali* and *Shadguna balijarita Rasasindhura* was 8 and 7 respectively, indicating mild alkaline nature of the sample.

Ash value

Ash value of *Shadguna Kajjali* was 0.13%

Ash value of *Shadguna balijarita Rasasindhura* was 0.04% respectively.

Acid insoluble ash

Acid insoluble ash value of *Shadguna Kajjali* was 0.02%. Acid insoluble ash of *Shadguna balijarita Rasasindhura* was 0.05%.

Loss on drying at 110°C

Loss on drying value of *Shadguna Kajjali, Shadguna balijarita rasasindur* were 0.23% and 0.04% respectively.

Zeta Potential Studies

In present study, *Shadguna balijarita Rasasindhura* (SDBRS) the Zeta potential values recorded was 22.23mv.

XRD

Both the *Rasasindhooraa* were identified as Cinnabar with Hexagonal crystal system having primitive lattice.

Highest peak count in *Shadguna balijarita Rasasindhura* was 1189.00, which indicates more crystallinity *Shadguna balijarita Rasasindhooraa* was identified as Cinnabar i.e., there is difference in intensity. Thus it can be inferred that there is definite difference in crystallinity in *Shadguna balijarita Rasasindhura* crystals.

Thus it can be considered that there was a difference in all the three i.e., Std Cinnabar, *Shadguna balijarita rasasindura* and XRD pattern.

When the particle size is more and the molecular aggression is rich, the crystallanity of a material is bound to increase. The crystallanity of *Shadguna balijarita Rasasindur* is small crystal size and is much finer and because of this reason the therapeutic activity of *Shadguna balijarita Rasasindur* is more potent.

Number of major peaks in *Shadguna balijarita Rasasindur* is more. It reflects that in *Shadguna balijarita Rasasindur* additional trace elements were found may be because of long duration of *Agnisamaskar* in *Shadguna balijarita Rasasindura,* thus resulted in additional peaks. Hence because of this reason *Shadguna balijarita Rasasindur* is more potent and *Yogvahi*.

EDAX

EDAX refers to energy dispersion X-ray spectroscopy and is employed here as an analysis tool to determine the elemental composition of *Shadguna balijarita Rasasindur*. It works by analyzing the spectrum of emitted x-rays from a sample as a beam of high energy electron is incident upon it. Two elements were observed in major concentration in the samples tested in *Shadguna balijarita Rasasindur.* They are S & Hg, S with weight% of 54.31% and atomic% of 88.15 % and Hg with weight% of 45.69% and atomic% of 11.85 respectively. The atomic% of Hg is less but its mass% is more because Hg is heavy metal.

CONCLUSION

In *Shadguna Kajjali*, free mercury was in trace levels, where as *Shadguna balijarita rasasindur* free mercury was nil, which proves the *Nischandratva* of *Kajjali* and *Rasasindura* and indicates that all procedures were properly carried out.

Percentage of total mercury in *Shadguna Kajjali* and *Shadguna balijarita rasasindur* was 40.03% and 84.17%.

Percentage of free sulfur in and *Shadguna Kajjali* was 43.07% respectively.

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***Address for correspondence**
Dr.Sorab Gaind
Lecturer
Department of Rasa Shastra,
Guru Nanak Ayurvedic Medical College and Hospital, Sri Muktsar Sahib, Punjab, India.
Email: dr.sorabgai@gmail.com