



Research Article

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STANDARDIZATION OF SURYA SHEKHARA RAS: A KUPI PAKWA RASAYANA

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ABSTRACT

Demand and acceptance of rasa aushadhis would not increase until unless these mineral and metallic preparations, particularly those which contain heavy metals like Surya Shekhara Ras are standardized. Surya Shekhara Ras is a typical herbo-mineral Kupipakwa rasayana indicated for fever especially in viral fevers. The present work highlights the procedures and techniques adopted for the physico-chemical analysis and characterization of three different samples of Surya Shekhara Ras obtained by well defined process in Rasa Shastra text. Pharmaceutically prepared Surya Shekhara Ras was subjected for physico-chemical analysis, X-ray diffraction (X-RD), Inductive Coupled Plasma with Optical Emission Spectrometry (I.C.P.O.E.S.), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Analysis (EDAX) and Fourier Transform Infrared Spectrometry (FT-IR) with a view to standardize the drug through modern techniques. Values of Physico-chemical studies of all three samples were almost in the same range. HgS compound form was detected in X-RD study of 3 samples with Hexagonal crystal structure. I.C.P.O.E.S showed presence of 94 % Hg. FT-IR analysis reveals the presence of similar organic functional groups in three samples of Surya Shekhara Ras. As final results and quantity of elements in all three samples of Surya Shekhara Ras were in same range, it reveals the uniformity in quality of prepared medicine and purity of drug. Thus suggests that the adopted method of preparation can be taken as standard.

Keywords: Kupipakwa Rasayana, Surya Shekhara Ras, Standardization, X-RD, ICPOES, SEM-EDAX, FTIR

INTRODUCTION

Kupipakwa Rasayana¹ is a unique pharmaceutical process where Parada (Mercury) processed with other drugs in Kach Kupi² (Glass bottle) with krama agni and through the preparation mercury gets potentially upgraded and drug attains Rasayana property. Surya Shekhara Ras³ is a saagni, sagandha moorchana of shuddha Parada⁴, shuddha Gandhaka⁵, shuddha Hingula⁶ and shuddha Vatsanabha⁷ (*Aconitum ferox*) together as main ingredients in the ratio of 3:4:1:8 respectively which are potentiated through krama agni for 18 h in Baluka yantra. WHO ensure the need of the quality control of Ayurvedic products by using modern techniques and by applying suitable standards. Therefore it is highly desirable that these drugs should be characterized with modern instruments which provide objective characteristics, based on which the specifications of such drugs can be well standardized on a scientific basis. Apart from this physico-chemical characterization like Total ash, Acid insoluble ash and Loss on drying etc. are needed to test the quality of drug. By observing the approximately equal values in all batches we can say that the quality of product is being maintained. The analytical methods reveal out the chemical composition of formulation as well as their concentration. These studies will help to ensure safety limits and accuracy of the drug.

MATERIAL AND METHODS

In this study following methods have been adopted for physicochemical characterization and standardization of Surya Shekhara Ras samples prepared in 3 batches.

Determination of Loss on Drying

It is the measure of the amount of water and volatile matters in a sample when the sample is dried at 110°C⁸.

Procedure

The instrument was calibrated to 0 deflections. 4 grams of accurately weighed sample was placed in the tarred pan inside the infra-red moisture balance. Thermostat was adjusted at 105°C. The sample was kept till the deflection shows a constant reading. Sample was taken out cooled and weighed. The loss of weight in mg per g. of the air-dried material was calculated.

Determination of pH Value

Method

pH meter and electrode system was operated according to the manual instructions. At the end of a set of measurements, reading of the solution used for standardizing the meter and electrodes was taken. This reading should not differ by more than 0.02 from the original value at which the apparatus was standardized. One gram sample was put in 5ml of water and pH of the solution was determined⁹.

Determination of Total Ash Value

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 the weight of the ash was obtained and the ash value of sample was calculated¹⁰.

Determination of Acid Insoluble Ash

2 g of sample was digested with 25 ml diluted hydrochloric acid for 5 minutes, then filtered through Whatman paper and washed with water. The residue was taken in a crucible dried and ignited, allowed to cool and weighed¹⁰.

Determination of Water Soluble Ash

Ash was boiled for 5 minutes with 25 ml of water. Insoluble matter was collected in an ashless filter paper; washed with hot water, and ignited for 15 minutes at a temperature not exceeding 450°C. Subtracted the weight of the insoluble matter from the weight of the ash; the difference in the weight represents the water soluble ash. Percentage of water soluble ash was calculated with reference to the air dried drug.

Physicochemical Analysis of Surya Shekhara Ras (Table 1)

Solubility Test

10 ml of each solvent was taken separately in a test-tube. Then 100 mg of each sample of Surya Shekhara Ras was added in the solvent. Then it was shaken for every 15 minutes up to 12 hours. After 12 hours, solubility was observed. (Table 2)

Powder X-Ray Diffraction (X-RD) Study

X-ray diffraction technique is the most useful in the characterization of crystalline materials such as minerals, metals, inter metallics and ceramics. It is used for qualitative and quantitative phase identification of crystalline, grain size, lattice parameters and identifying different crystal structures of the compound. The basic principle of the phase analysis using powder XRD technique lies the presence of diffraction peaks corresponding to various inter planar (d_{hkl}) spacing which are characteristics of a given material. The relative intensities of various peaks occurring at different d-spacing are also different for different phases.

Analysis Procedure

The sample preparation for the analysis was done using standard XRD procedure. The powder was then spread onto a double-side tape with a spatula, and then placed on a cavity with plastic holder. Care was taken to fill the powder uniformly into the mount. It was exposed to x-ray beam of intensity 35 KV and 20 MA. All the peaks were recorded on the chart, and the corresponding 2-theta values were calculated.

Instrument Specification: Philips X-ray generator PW 3020 attached with graphic mono chromator

Inductively Coupled Plasma with Optical Emission Spectroscopy (ICPOES)

The method is suited for multi-element determination in solutions or after appropriate sample preparation of solid samples brought into solution. Measurement time is generally a few minutes per sample. The ICP-OES used in the study is Perkin Elmer Optima 4300 V (Simultaneous) based on Argon plasma

Place of ICP-OES Study: Analytical Chemistry Dept., D.M.R.L., Hyderabad, India.

Scanning Electron Microscope

SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. SEM also produces images of high resolution.

Analysis Procedure

SEM of the sample was analyzed by S-2400 Hitachi Scanning Electron Microscope. To produce the SEM image, the electron beam was swept across the area being inspected, producing many such signals. These signals are then amplified, analyzed and translated into images of the topography being inspected. Finally, the image is shown on a CRT.

Place of Study: SEM Dept. D.M.R.L. Hyderabad, India

Energy Dispersive X-Ray Analysis (EDAX)

A Scanning Electron Microscopy (SEM) may be equipped with an EDAX analysis system to enable it to perform compositional analysis on specimens. EDAX analysis is useful in identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen.

Place of Study: D.M.R.L, Hyderabad, India

Fourier Transform Infrared Spectrometry (FT-IR)

In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some are passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular finger prints of the sample. Like a fingerprint, no two unique molecular structures produce the same infrared spectrum. It can identify unknown materials, determine the quality or consistency of a sample and determine the amount of components in a mixture. The FTIR spectra of the samples were recorded between GX Range: 30 - 15600 cm^{-1} using Perkin Elmer Spectrum with ATR accessory for reflectance measurement; IR Quant software; Spectrum search software.

RESULTS

X-RD Study

Surya Shekhara Ras I (Figure 1)

Several Peaks of HgS (Cinnabar) compound were present. No other compound was detected. The strongest peak was present at 2-theta Scale at 26-27 and 45-46. Crystal structure was Hexagonal.

Table 1: Result of physico-chemical analysis of Surya Shekhara Ras

S. No	Test	Surya Shekhara Rasa- Batch I	Surya Shekhara Rasa –Batch II	Surya Shekhara Rasa – Batch III
1.	Loss on drying	0.12 %	0.14 %	0.12 %
2.	pH	5.75 + 0.10	5.79 + 0.10	5.61 + 0.10
3.	Total Ash Value	12.22 %	12.00 %	12.24 %
4.	Acid insoluble ash	3.15 %	3.24 %	3.11 %
5.	Water soluble ash	0.30	0.34	0.32

Table 2: Solubility tests of Surya Shekhara Ras

Name of the Solvent	Solubility		
	Surya Shekhara Ras –I	Surya Shekhara Ras –II	SuryaShekhara Ras –III
Water, Methanol, Toluene	Insoluble	Insoluble	Insoluble
Chloroform, Ethyl alcohol	Slightly soluble	Slightly soluble	Slightly soluble
Hydrochloric acid	Insoluble	Insoluble	Insoluble

Table 3: Result of ICP-OES of Surya Shekhara Ras

Sample Name	Hg (%) (ppm)	Fe (ppm)	Sn (ppm)	Mg (ppm)	Ca (ppm)
Surya Shekhara Ras (I)	94.12	Nil	Nil	Nil	Nil
Surya Shekhara Ras (II)	94.14	Nil	Nil	Nil	Nil
Surya Shekhara Ras (III)	94.11	Nil	Nil	Nil	Nil

Table 4: Compositions of Surya Shekhara Ras I with elemental % by EDAX

Elmt	Spect.Type	Element %	Atomic %
O K	ED	2.18	12.79
Al K	ED	0.44	1.54
S K	ED	16.32	47.75
Hg L	ED	81.06	37.92
Total		100.00	100.00

Table 5: Compositions of Surya Shekhara Ras II with elemental % by EDAX

Elmt	Spect.Type	Element %	Atomic %
O K	ED	2.33	13.43
Al K	ED	0.69	2.36
S K	ED	16.35	47.09
Hg L	ED	80.63	37.12
Total		100.00	100.00

Table 6: Compositions of Surya Shekhara Ras III with elemental % by EDAX

Elmt	Spect.Type	Element %	Atomic %
O K	ED	2.38	12.71
Al K	ED	0.46	1.56
S K	ED	16.12	47.75
Hg L	ED	81.04	37.98
Total		100.00	100.00

Table 7: FT-IR Observations of Surya Shekhara Ras I

Absorption peak Frequency Cm^{-1}	Standard peaks Frequency Cm^{-1}	Specific Type of bond	Bond	Functional Group
3435	3500-3200	Strong	N-H	Primary Amines
2920	3000-2850	Multiple Broad Peaks	N-H	SecondaryAmines
2850	3000-2850	Multiple Broad Peaks	N-H	SecondaryAmines
2103	2260-2100	Weak	-C=C-	Alkynes
1632	1650-1585	Strong	N-H	Alkanes
1456	1500-1400	Weak /Strong	C=C	Aromatic
1120	1320-1000	Weak / Strong	C=O	Ethers, Alcohol
618	850-550	Weak / Medium	-C-X-	Chloralkanes

Table 8: FT-IR Observations of Surya Shekhara Ras II

Absorption peak Frequency Cm^{-1}	Standard peaks Frequency Cm^{-1}	Specific Type of bond	Bond	Functional Group
3432	3500-3200	Strong	N-H	Primary Amines
2922	2870-2960	Medium/ Strong	C-H	Alkanes,Methylane
2855	2850-2925	Medium/Strong	C-H	Alkanes,Methylane
2100	2260-2100	Weak	C=C	Alkynes
1630	1560-1640	Strong	N-H	Alkanes
1405	1500-1400	Weak/Strong	C=C	Aromatic
1115	1100-1200	2 Strong bands	C-X	Fluroalkanes
614	540-760	Medium /Strong	C=C-H	Bromo-alkanes

Table 9: FT-IR Observations of Surya Shekhara Ras III

Absorption peak Frequency Cm^{-1}	Standard peaks Frequency Cm^{-1}	Specific Type of bond	Bond	Functional Group
3436	3500-3200	Strong	N-H	Primary Amines
2925	2870-2960	Medium/Strong	C-H	Alkanes, Methylane
2850	2850-3000	Weak /Strong	C-H	Alkanes
2062	2850-2925	Weak	C=C	Alkynes
1630	1560-1640	Strong	N-H	Alkanes
1385	1500-1400	Weak /Strong	C=C	Aromatic
1051	1100-1200	Ordinary	C-X	Fluroalkanes
670	540-760	Medium/Strong	C=C-H	Chloroalkanes

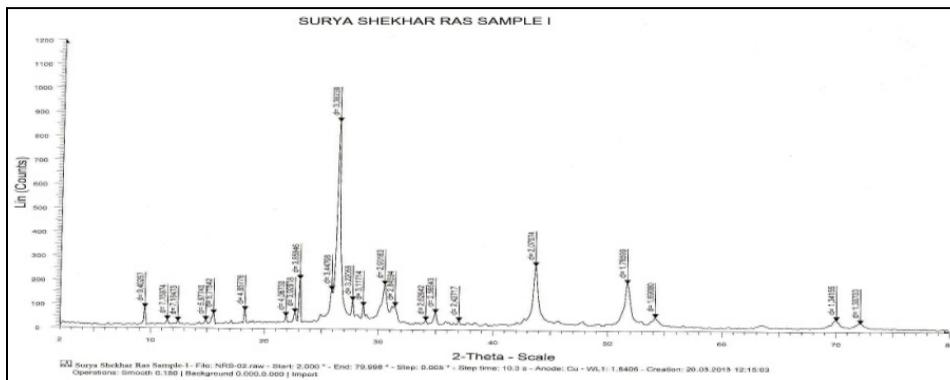


Figure 1: X-RD Pattern of Surya Shekhara Ras I

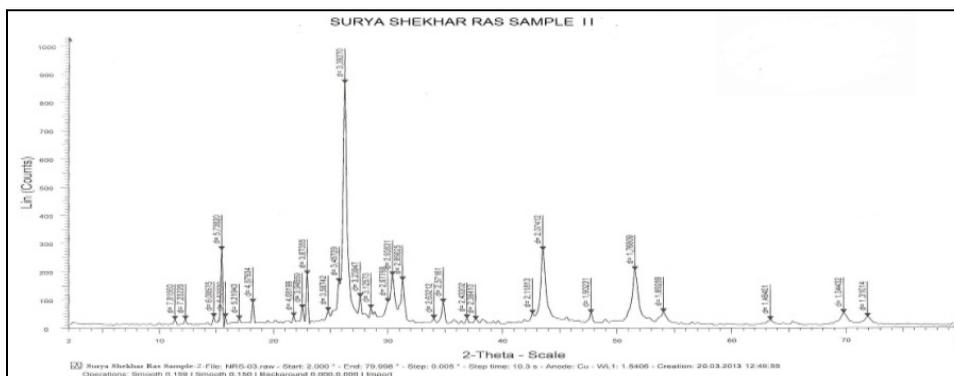


Figure 2: X-RD Pattern of Surya Shekhara Ras II

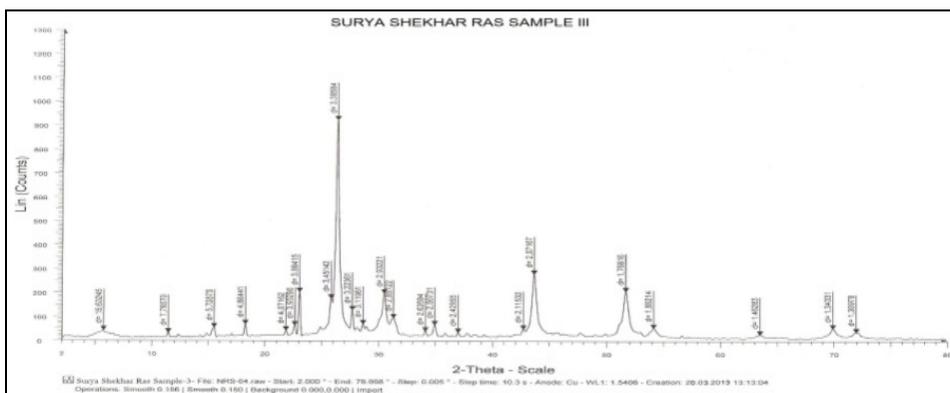


Figure 3: X-RD Pattern of Surya Shekhara Ras III

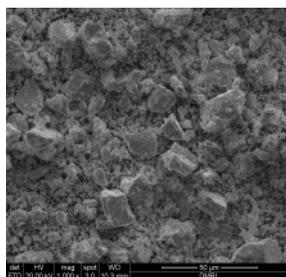


Figure 4: SEM picture of Surya Shekhara Ras-1000X

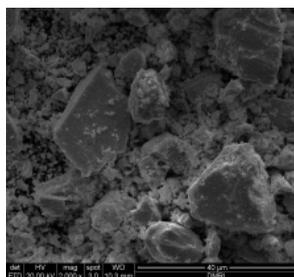


Figure 5: SEM picture of Surya Shekhara Ras-2000X

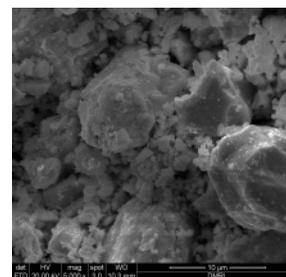


Figure 6: SEM picture of Surya Shekhara Ras - 5000X

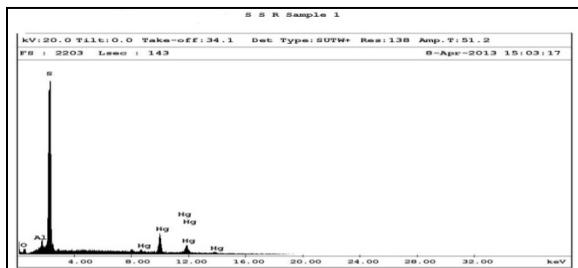


Figure 7: EDAX of Surya Shekhara Ras I

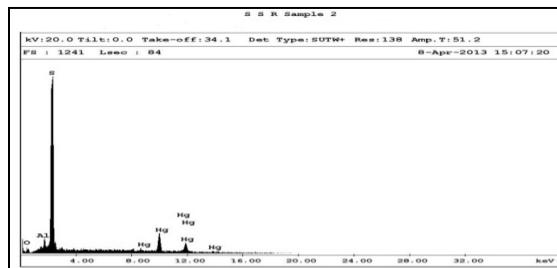


Figure 8: EDAX of Surya Shekhara Ras II

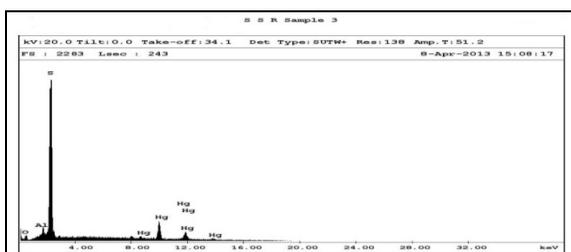


Figure 9: EDAX of Surya Shekhara Ras III

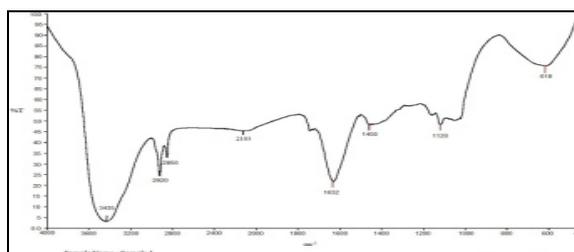


Figure 10: FT-IR of Surya Shekhara Ras I

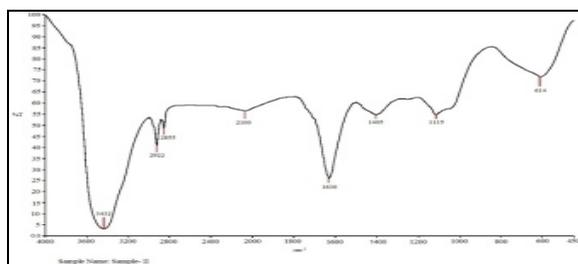


Figure 11: FT-IR of Surya Shekhara Ras II

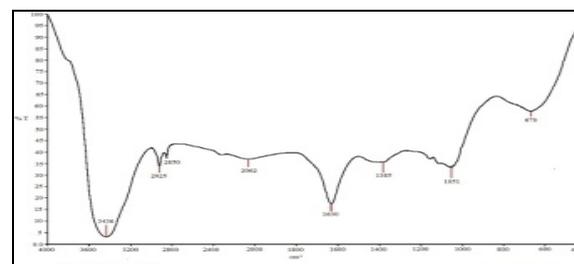


Figure 12: FT-IR of Surya Shekhara Ras III

Surya Shekhara Ras II (Figure 2)

HgS (Cinnabar) compound was present. No other compound was detected. The strongest peak was present at 2-theta Scale at 26-27 and 44-45. Crystal structure was Hexagonal.

Surya Shekhara Ras III (Figure 3)

HgS (Cinnabar) compound was present. No other compound was detected. The strongest peak was present at 2-theta Scale at 26-27 and 44-45. Crystal structure was Hexagonal.

ICPOES Study (Table 3)

SEM Observation: Spongy, relatively compact microcrystalline aggregates with loss of grain boundaries were observed in all samples. (Figure 4-6)

EDAX Study of Surya Shekhara Ras (Figure 7-9)

EDAX shows both the Elemental and Atomic percentage of the elements which are present in the drug. As per EDAX study elemental percentage in Surya Shekhara Ras- I (Table 4) is Hg - 81.06 %, S - 16.32 %, O - 2.18 %, Al - 0.44 %, In Surya Shekhara Ras -II (Table 5) is Hg - 80.63 %, S - 16.35 %, O - 2.33 %, Al - 0.69 % and Surya

Shekhara Ras III (Table 6) is Hg - 81.04 %, S-16.12 %, O - 2.38 %, Al - 0.46 %.

FT-IR Analysis gave eight notable absorption peaks in all samples. FTIR spectrum of SSR I (Figure 10) gave peaks at 618- 3435 cm^{-1} , SSR II (Figure 11) at 614-3432 cm^{-1} and SSR III (Figure 12) at 670- 3436 cm^{-1} .

SUMMARY AND CONCLUSION

Three samples of Suryashekhara Ras prepared in 3 different batches have been subjected to Physico-chemical analysis for pharmaceutical standardization of the drug. Elemental composition and characterization of all the samples have given almost same results. According to X-RD Report of 3 drug samples, all peaks of HgS were found at almost same 2 theta degrees with Hexagonal crystal structure. ICPOES showed presence of 94.12 %, 94.14 %, and 94.11 % of Hg in sample I, II, III respectively. The Scanning Electron Microscopic studies shows spongy relatively compact microcrystalline aggregates with loss of grain boundaries in all samples. The elements present in 3 samples of Surya Shekhara Rasa are: Mercury, Sulphur, Oxygen, and Aluminum. EDAX shows absence of heavy metals Cadmium, Lead and Tin. FT-IR analysis reveals the presence of similar organic functional groups in all three samples (Table 7-9). Analytical study of different samples of Surya Shekhara Ras has justified different processes followed during the pharmaceutical preparation of these samples, as variations in the values are very less and as per line of pharmaceutical process.

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