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Chemical Characterization of Samaguna Balijarita Kajjali (Black Sulphide of Mercury)

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ABSTRACT
Kajjali (Black Sulphide of Mercury) is a Sagandha (together with sulphur), Niragni (fire is not involved in the process) Parada yoga (mercurial preparation). The Bandha (bond) involved in this preparation is Kajjali Bandha, where purified Parada (mercury) and Gandhaka (sulphur) are thoroughly ground in definite proportion, to get a fine black powder called Kajjali. Among Khalviya rasayana (mortar pestle apparatus used medicinal preparations), Kajjali is having prime importance as it forms base to many mercurial preparations. Though Ancient scholars mentioned some quality checks and end points in preparing Bhasma like Varitaratva, Rekhapurnatva, etc., these are not sufficient in the present scenario. Apart from these testing procedures, there is need of more sophisticated testing methods for determining the quality of Kajjali. The Present work illustrates the chemical characteristics of Kajjali obtained by well-defined process in the Ayurvedic classics. This Kajjali was evaluated by Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Analysis (EDAX). SEM images with the magnifications of 2.00KX, 1.00 KX and spatial resolution of 25 nm represented that it formed the order of agglomerates consisted of small spherical particles approximately size of 3 µm to 10 µm observed, whereas EDAX showed the presence of Hg (69.33%), S (24.44%), O (4.64%) and Ag (1.59%) in the final preparation of Kajjali.

Key Words: Kajjali, Black Sulphide of Mercury, Scanning Electron Microscope (SEM), Energy Dispersive X-Ray Analysis (EDAX), Mercury (Hg), Sulphur (S)

INTRODUCTION
Purified Parada (mercury) and Gandhaka (sulphur) ground without adding any liquid till it becomes a homogenous mixture and it looks like Kajjala (collyrium, a black soft substance which is applied over the edges of eyelids), is called Kajjali[1]. Kajjali is added with different Parada yoga (mercurial formulations) and such formulations are also called as Kajjali bandha[2]. Kajjali can be prepared by adding Gandhaka to Parada by half, equal, double quantities[3] etc. Internal administration of Kajjali cures many disorders, pacifies the Tridosha (disorder of the three humours of the body) and acts as Vrushya (an aphrodisiac). Further, it is also used as Sahapana (taking together with the medicine) and Anupana (a vehicle taken after the medicine)[4].
In addition to above, kajjali is also one of the prime ingredient in various Rasayoga (herbo-mineral formulations)[5] and is used as a medicine separately. There are different proportions of purified Parada and Gandhaka are mentioned in the preparation of Kajjali.The present work was aimed at preparation and chemical characterisation of Samagunabali Jarita Kajjali (equal parts of mercury and sulphur).

MATERIALS AND METHODS

Material
The ingredients were procured from the local market of Vijayawada, Andhra Pradesh, India and all the herbal and mineral material were thoroughly screened by experts of P.G. Dept. of Rasashastra, Dr.N.R.S.Govt.Ayurvedic College, Vijayawada, AP., based on the Grahya lakshanas (to be taken or accepted characters) mentioned in the classics.

Purification of the Ingredients

A. Purification of Parada (Mercury)
Parada (250g) is taken with Nagavalli svarasa (50ml of juice of betel leaf), Ardraka svarasa (50ml of ginger juice) and Trikshara (50g each of alkalies of Yava, Sarja, Tankana) in a clean Khalva yantra (mortar and pestle) and grind eight hours daily for three days[6]. The obtained material was washed with the help of lukewarm water for several times until clean and clear Parada is obtained. Approximately 230g Parada was obtained after this purification process. Fig. 1 shows the purification steps of Parada.

B. Purification of Gandhaka (Sulphur)

A wide mouth Ghata (earthen vessel) is taken and filled with 2 litres of Gokshira (Cow's milk) and 150 ml of Goghrita (Cow's ghee) then the mouth of vessel is covered by a cloth and tied by iron wire. Coarse powder of Gandhaka (500g) spread upon the cloth and closed with another earthen vessel by placing in up-down position. The edges of both the vessels are sealed with Multani mitti (Fuller's earth.) smeared cloth for ten times and allowed it to dry under sunlight. This Yantra (apparatus) is kept inside a pit (1.5 feet) beneath the surface of soil in such a way that, the brim of the vessel should be at ground level. Empty space of the pit around the apparatus was filled by soil. Cow dung cakes were kept on above said brim of the vessel and set on fire. The Sulphur, after melting by fire flows down through cloth into the vessel, which contained milk and ghee. After Svangasita (self-cooling), the apparatus is taken out from the pit and Sandhibandhana (seal) was opened carefully. Purified Gandhaka (470g, in granules form) was collected from the bottom of vessel and washed with hot water[7]. Fig. 2 shows the purification steps of Gandhaka.

Preparation of the Kajjali

Purified Parada and Gandhaka were taken in equal quantities (250g each) in a Khalva yantra and Mardhana (grinding) was done until it becomes very fine black powder, like collyrium and the dazzling particles of Mercury completely disappeared[8]. Approximately 470g of Kajjali was obtained. Fig. 3 shows the final prepared Kajjali.

Characterization of the Kajjali

1) Scanning Electron Microscope

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). The design and function of the SEM is very similar to the EPMA and considerable overlap in capabilities exists between the two instruments[9].

During SEM inspection, a beam of electrons is focused on a spot volume of the specimen, resulting in the transfer of energy to the spot. These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attached and collected by a positively biased grid or detector, and then translated into a signal. To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analysed, and translated into images of the topography being inspected. Finally, the image is shown on a CRT.

Evaluation of Sample

The fine powder of Kajjali was placed inside the microscope's vacuum column through an airtight door, and then the air was pumped out. After the air was pumped out of the column, a beam of electrons was emitted by an electron gun from the top. This beam travels downward through a series of magnetic lenses designed to focus the electrons to a very fine spot. Near the bottom, a set of scanning coils made the focused beam to move back and forth across the mounted sample, row by row.

As the electron beam hits each spot on the sample, secondary electrons are backscattered from its surface. A detector counts these electrons and sends the signals to an amplifier. The microscopic photographic image was built up from the number of electrons emitted from each spot on the sample.

2) Energy Dispersive X-Ray Analysis

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

This technique is used in conjunction with SEM and is not a surface science technique. An electron beam strikes the surface of a conducting sample. The energy of the beam is typically in the range 10-20keV. This causes x-rays to be emitted from the point the material. The energy of the x-rays emitted depends on the material under examination. The x-rays are generated in a region about 2 microns in depth, and thus EDX is not a surface science technique. By moving the electron beam across the material an image of each element in the sample can be acquired in a manner similar to SAM. Due to the low x-ray intensity, images usually take a number of hours to acquire. Elements of low atomic number are difficult to detect by EDX. The SiLi detector (see below) is often protected by a Beryllium window. The absorption of the soft x-rays by the Be precludes the detection of elements below an atomic number of 11(Na). In windowless systems, elements with as low atomic number as 4 (Be) have been detected, but the problems involved get progressively worse as the atomic number is reduced.

The Lithium drifted Silicon (SiLi) detector

The detector used in EDX is the Lithium drifted Silicon detector. This detector must be operated at liquid nitrogen temperatures. When an x-ray strikes the detector, it will generate a photoelectron within the body of the Si. As this photoelectron travels through the Si, it generates electron-hole pairs. The electrons and holes are attached to opposite ends of the detector with the aid of a strong electric field. The size of the current pulse thus generated depends on the number of electron-hole pairs created, which in turn depends on the energy of the incoming x-ray. Thus, an x-ray spectrum can be acquired giving information on the elemental composition of the material under examination.

RESULTS AND DISCUSSION

The SEM pictures with 2.00 KX and 1.00 KX resolution of 25 nm are taken for the samples. The surface of the Kajjali grains is irregular and sharp, it seems to develop first at exposed edges and corners of surfaces approximately size of 3 µm to 10 µm observed in fig. 4 and 5.

In the Kajjali metal Hg used in the other then sulphur, EDAX has been used to detect the elements present a considerable trace amount. Table-1 shows chemical compositions of Kajjali using EDAX (See fig. 6). The trace metal composition for Mercury, Sulphur, Oxygen and Silver was found to be 69.33%, 24.44%, 4.64% and 1.59% respectively.

During the EDX analysis of Kajjali surprisingly there was 1.59% of elemental Silver available along with the Mercury and Sulphur.

CONCLUSION

The Present work illustrates the chemical characteristics of Kajjali obtained by the well-defined processes in the Rasashastra text, which is prepared with the equal quantities of purified Parada and Gandhaka. Further Kajjali is subjected to SEM, EDAX analytical instruments. The details including results of these testing procedures with illustrated photographs, tables etc. are mentioned in the paper. Scanning Electron Microscopy (SEM) images represents with the magnifications of 2.00 KX, 1.00 KX and spatial resolution of 25nm was seen that it formed the order of agglomerates consisted of small spherical particles approximately size of 3 µm to 10 µm observed. It is this significant size which allows the phenomenon of Rekhapurna and Varitara to develop,whereas EDAX showed the presence of Hg (69.33%), S (24.44%), O (4.64%) and Ag (1.59%) in the finally prepared Kajjali.

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Table-1: Element Composition of Kajjali (Samaguna Balijarita)

Standards:

O K	Quartz 01/12/ 93
S K	FeS_2 01/12/ 93
Ag L	Ag 01/12/93
Hg M	HgTe 01/12/93

Results:

Elements	Spect. Type	Element %	Atomic %
O K	ED	4.64	20.54
S K	ED	24.44	53.96
Ag L	ED	1.59	1.04
Hg M	ED	69.33	24.47
Total		100.00	100.00

*=<2 Sigma

Fig.1: Purification of Parada (mercury)

Fig.2: Purification of Gandhaka (sulphur)

Fig.3: Prepared Kajjali

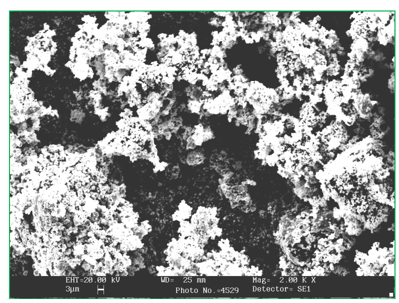

Fig.4: SEM image of Kajjali 1.00 KX

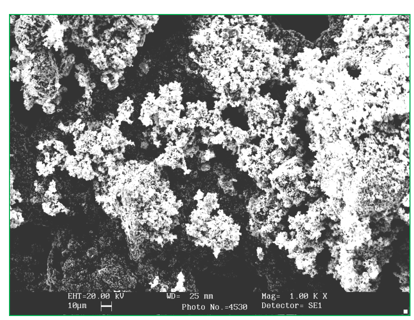

Fig.5: SEM image of Kajjali 2.00 KX

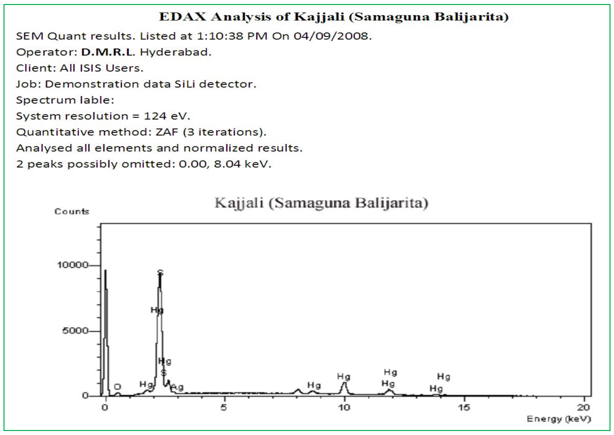

Fig.6: EDAX of Kajjali

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