



# PHARMACEUTICO-ANALYTICAL STUDY OF SILATAALARASA

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## ABSTRACT

Herbal and herbo-mineral medicines have been widely utilized in Ayurveda. Ayurvedic medicines have indications that span a broad spectrum of health concerns. Among these *Rasaoushadhi* holds a significant place in Ayurveda due to its remarkable rejuvenating qualities and potent efficacy even at low doses. *Silataalarasa* is an herbo-mineral formulation explained in *Brihat Rasaraja Sundhara Kasaadhikara*. It contains *Haratala* (Orpiment), *Manasila* (Realgar), *Shunti*, *Maricha*, *Pippali*, *Nirguntimoola*, *Vasa* and *Gokshura*. This study aimed to prepare *Silataalarasa* using the classical Kupipakwa method, analyzing its physical-chemical properties and compositional characteristics. Three batches of *Silataalarasa* were meticulously prepared, adhering to the traditional methodology outlined in ancient texts. Pharmaceutico-analytical investigations revealed crucial parameters: LOD 5.31%, Total Ash 5.04%, pH 5.41 and acid insoluble Ash 0.91 %. High-Performance Thin-Layer Chromatography (HPTLC) offered insights into compound composition and distribution within the formulation. SEM-EDS shows the formulation is of micro size and mostly organic matters. XRD analysis shows that this formulation contains  $As_4S_4$ . The data obtained from pharmaceutical, analytical, and instrumental studies collectively contribute to a preliminary profile for *Silataalarasa*.

**Keywords:** *Silataalarasa*, *kupipakwa*, *pharmaceutico analytical study*

## INTRODUCTION

Throughout history, diverse system of medicines has emerged worldwide as responses to the enduring human quest for optimal health. *Ayurveda* is also included within these various medicinal systems. *Ayurveda* offers a comprehensive approach to health and wellbeing. *Rasasastra* is an important branch of *Ayurveda* which deals with the use of metallics and mineral drugs. Within the realm of *Ayurveda*'s multifaceted concepts, the prominence of *Rasasastra* shines through, offering invaluable contributions to the management of a wide array of illnesses. *Rasasastra* began with '*Dhatu vada*', which later transformed into the concept of '*Dehavada*'. This evolution primarily revolved around a practical approach to converting metals and minerals into compounds suitable for therapeutic use.

*Rasaoushadhis* have special attributes like lesser therapeutic dose, quicker action and palatability<sup>1</sup>. So herbo- mineral formulations are very potent in nature. Hence this system of medicine is superior compared to vanaspati yogas<sup>2</sup>. *Silataalarasa* is a simple herbo mineral formulation mentioned in *Brihat Rasaraja sundara Kasaadhikara*<sup>3</sup> which is prepared by using *Haratala*, *Manasila*, *Trikatu*, *Nirgundi*, *Vasa swarasa*, *gokshurarasa*. It is indicated in *Swasa* and *Kasa*. *Swasa* and *Kasa* in *Ayurveda* is mentioned in a broad aspect and wide variety of respiratory diseases Most of the ingredients present in this formulation have proven its anti-microbial activity. *Haratala* and *Manasila* are commonly used in treating the diseases like *Sleshmaroga*, *Kasa*, *Swasa*, *Kushta*<sup>4,5</sup>.

Respiratory diseases, whether acute or chronic, communicable or non-communicable impose a major global and affects millions of people. According WHO, respiratory diseases are leading causes of death and disability in the world. Approximately 65 million people suffer from acute respiratory tract infections and three million dies from them each year. These infections are the third leading cause of death all over the world<sup>6</sup>. In present scenario, chronic respiratory diseases are common among patients with COVID-19.

Hence, an effort has been made to prepare *Silataalarasa* following the guidelines of the *Brihat Rasaraja Sundara* and also analysing the prepared medicine as part of a research protocol to understand its composition and potential benefits for society's improvement.

## AIM AND OBJECTIVE

This study is an attempt to prepare *Silataalarasa* and to develop analytical profile of *Silataalarasa*.

## MATERIALS AND METHODS

### Review of Literature

- All the classical reference and contemporary literature of *Silataalarasa* will be reviewed.
- The present reference of the study of *Silataalarasa* is from *Brihatrasaraja sundara Kasa adhikara*

### Selection and collection of raw drugs

Mineral drugs such as *Haratala* and *Manasila* were collected from a retailer in Madurai. The authenticity was confirmed based on *Grahya Lakshanas* mentioned in the *Ayurvedic* classics, and modern parameters were also assessed. Subsequently, ideal samples preferred for this study were selected. In the case of herbal drugs, samples were collected from both the local market and also from the herbal garden of the college.

Three batches of *Silataalarasa* were prepared for the analytical purpose in the Department of Rasasastra and Bhaishajya Kalpana, laboratory, MVR Ayurveda Medical college, Parassinikkadavu.

**Table No: 1 Table showing ingredients and composition of *Silataalarasa***

| Sl No | Ingredients           | Botanical name                            | Parts used   | Classical Quantity  |
|-------|-----------------------|---|--------------|---|
| 1     | <i>Haratala</i>       | As <sub>2</sub> S <sub>3</sub> , Orpiment | Mineral drug | 1 part  |
| 2     | <i>Manasila</i>       | As <sub>2</sub> S <sub>2</sub> , Realgar  | Mineral drug | 4 parts   |
| 3     | <i>Gokshura</i>       | <i>Tribulus terrestris</i>                | Fruit        | For <i>Bhavana</i>  |
| 4     | <i>Vasa</i>           | <i>Adhatoda vasica</i>                    | Leaf         | For <i>Bhavana</i>  |
| 5     | <i>Pippali</i>        | <i>Piper longum</i>                       | Fruit        | Equivalent to the product obtained through <i>Kupipakwa</i> . |
| 6     | <i>Maricha</i>        | <i>Piper nigrum</i>                       | Fruit        |   |
| 7     | <i>Shunti</i>         | <i>Zingiber officinale</i>                | Rhizome      | Equal to quantity of <i>Trikatu</i>                           |
| 8     | <i>Nirgundi moola</i> | <i>Vitex negundo</i>                      | Root         |   |

## METHOD OF PREPARATION

- Shodhana of *Manasila*
- Shodhana of *Haratala*
- *Bhavana* of mixture of *Haratala* and *Manasila* with *Gokshura Swarasa* and *Vasa Swarasa*
- Preparation of *Silataala rasa*

### 1. *Manasila Shodhana*

Method of *Shodhana*: *Bhavana*<sup>7</sup>

Equipments: *Khalwa Yantra*, Spoon, measuring jar, Weighing Machine

**Table No: 2 showing ingredients for *Manashila Shodhana***

| SL NO. | DRUGS                     | QUANTITY |
|--------|---------------------------|----------|
| 1      | <i>Ashodhita Manasila</i> | 500g     |
| 2      | <i>Ardraka Swarasa</i>    | 360ml    |

### Procedure for *Manasila Sodhana*

Powder *Asodhita Manasila* using a clean *Khalwa Yantra*. Remove stone pieces if any by hand picking. Weigh 500g and add into *Khalwa Yantra* and made into fine powder. Add sufficient quantity of *Ardraka Swarasa* so that the powder is *Sarvam plutam*. Triturate the mixture till it turns dry completely. Repeat the procedure six more times. Collect the *Sodhita Manasila*, dry it under Sun light, powder it and pack it in air tight container.

### Observations

Colour of raw *Manasila* Sample- Reddish orange

Colour of fine powder of *Manasila*- Reddish orange

Weight of *Sodhita Manasila* after drying- 550g

**Precautions:**

Clean *Khalwa Yantra* should be used.

Fresh *Ardraka Swarasa* should be used for each *Bhavana*.

**Table No: 3 observations of *Manasila***

| SL No | Particularis                                |             |
|-------|---|-------------|
| 1     | Amount of <i>Manasila</i> taken             | 500g        |
| 2     | Amount of <i>Sodhita Manasila</i> obtained  | 550g        |
| 3     | Amount of <i>Ardraka Swarasa</i> used       | 360 ml      |
| 4     | Total time taken to complete <i>Sodhana</i> | 21hr 30min  |
| 5     | Colour of <i>Sodhita Manasila</i>           | Dark Orange |
| 6     | Gain of weight                              | 50g         |

**2. *Haratala Shodhana***

Method of *Sodhana*: *dolayantra swedana*<sup>8</sup>

Equipments: *Khalwa Yantra*, Spoon, measuring jar, Weighing Machine, mud pot

**Procedure**

*Asodhita Haratala* is taken in a cloth. Remove stone pieces if any by hand picking and made into a pottali. Add sufficient quantity of *Kushmanda swarasa*. Subjected to *Swedana* for 3 hours. Collect the *Sodhita Haratala*, dry it under Sun light, powder it and pack it in air tight container.

**Table No: 4 Observations of *Haratala Shodhana***

| Sl No. | Particularis                                |               |
|--------|---|---------------|
| 1      | Amount of <i>haratala</i> taken             | 500g          |
| 2      | Amount of <i>shodhita haratala</i> obtained | 495g          |
| 3      | Amount of <i>kushmanda swarasa</i> used     | 3500ml        |
| 4      | Time to complete <i>shodhana</i>            | 3 hours       |
| 5      | Colour of <i>shodhita haratala</i>          | Yellow Orange |
| 6      | Loss of weight                              | 5g            |

**3. *Bhavana* of mixture of *Haratala* and *Manasila* with *Gokshura Swarasa* and *Vasa Swarasa*****Table No: 5 Showing observations of *Bhavana***

|   |                         | Sample 1             | Sample 2             | Sample 3             |
|---|-------------------------|----------------------|----------------------|----------------------|
| Amount of <i>Manasila</i> taken         |                         | 40 g                 | 40 g                 | 40 g                 |
| Amount of <i>Haratala</i> taken         |                         | 10 g                 | 10 g                 | 10 g                 |
| Weight after <i>Bhavana</i>             |                         | 51 g                 | 53 g                 | 54 g                 |
| Consistency                             |                         | Powder form          | Powder form          | Powder form          |
| Colour                                  |                         | Orange               | Orange               | Orange               |
| Touch                                   |                         | Soft                 | Soft                 | Soft                 |
| Smell                                   |                         | Characteristic odour | Characteristic odour | Characteristic odour |
| Quantity of <i>Bhavana dravyas</i> used | <i>Gokshura Kashaya</i> | 180 ml               | 170 ml               | 175 ml               |
|   | <i>Vasa swarasa</i>     | 175 ml               | 175 ml               | 160 ml               |

#### 4. Preparation of *Silataala rasa*

##### Equipments required:

*Valuka Yantra, Kachakupi, Kora Cloth, Gopichandana, Cork, Iron Rods, Torch, Funnel, Infrared Gun, Kerosene, Thread, Khalwa Yantra, Gas Stove, copper coin, spatula, weighing machine.*

**Table No: 6 Showing ingredients of *Silataalarasa***

| INGREDIENTS AND QUANTITY                        | QUANTITY TAKEN |          |          |
|---|----------------|----------|----------|
|   | Sample 1       | Sample 2 | Sample 3 |
| Bhavitha <i>Manasila &amp; Haratala mixture</i> | 51 g           | 53 g     | 54 g     |

##### Procedure

The whole procedure was divided into 3 phases:

1. *Poorva Karma*
2. *Pradhana Karma*
3. *Paschat Karma*

##### Poorva Karma

The following steps were done:

- Collection of essential equipments and ingredients
- *Shodhana* of *Haratala* and *Manasila*.
- *Bhavana* of *Haratala* and *Manasila* with *Gokshura & Vasa swarasa*.
- iron rods are collected and cleaned for the purpose of *Salakas*.
- A torch is prepared for the proper visibility of inside of the *Kachakupi*.
- Selection of *Kachakupi*.
  - Mouth: should be narrow.
  - Neck: neither long nor short.
  - Colour: Amber colour
  - Should be transparent

- Selection of Mud pot

##### Preparation of *Kachakupi*

An elongated necked glass bottle of approximately 330ml capacity is taken and cleaned well. The first layer of cloth smeared with *Gopichandana* were applied around the *Kachakupi* and kept for drying under sunlight. After complete drying another 6 layers of cloth were applied and that too dried under sunlight.

##### *Kupipoorana*

The *Bhavitha Haratala & Manasila* mixture was cautiously added into the prepared *Kachakupi* using a funnel. 50g of mixture was filed in it and the remaining part is left vacant to allow better compound formation.

##### *Kupisthapana*

2-inch thickness of sand layer was made at the bottom of the earthen pot. Then the *Kupi* containing the drug was placed exactly at the centre of the *Valuka yantra*. The mouth of the *Kupi* was closed temporarily by cork and the earthen pot was filled up with *Valuka* up to the mouth level of the *Kupi*.

Total kg of sand was taken to fill the whole *Valuka yantra*.

##### Pradhana Karma

- *Agni* was given by using gas stove, *Mridu, Madhyama* and *Tikshna Agni* was given by adjusting the gas stove regulator. Heat was gradually increased.
- The cork of the bottle was removed soon after beginning the heating process.
- By using an infrared gun temperature was noted for every 15 minutes.
- During the procedure vapours along with fumes were expelled out from the mouth of the *Kupi* and the changes were noted.
- When fumes were reduced *Sheeta Salaka* was introduced into the *Kupi* which had got the blackish coloured coating of the product.
- Heat it for 6 hours then cork of the bottle was re-fitted and correctly sealed with mud smeared cloth. The *Valuka* around the neck of the bottle is removed and allowed to cool on its own.

##### Paschat Karma

- After cooled by its own, the bottle was taken out from the *Valuka yantra* and the mud and the cloth covering were carefully removed and bottle was cleaned from outside.

- After spotting the presence of the end product in the bottle, it was carefully wrecked by tying the kerosene-soaked thread around the bottle at accurate place and ignited and later on rolled the bottle in a wet cloth. Thus, the *Kupi* was broken exactly at the level of the thread.
- The product was seen at the base portion, a shiny blackish material was found.
- Shiny yellowish material was seen at the neck portion of the bottle.
- The prepared medicine at the base portion was carefully collected in the same shape by gently stroking the part of the bottle held inverted.
- It was checked whether the medicine contain any glass pieces mixed with it and later it was triturated in a porcelain *Khalwa yantra* to obtain fine powder of the same.

### Precautions

- Filling of ingredients should be done carefully.
- Sand should be clean and filtered before use. The sand was filled upto 3cm using 1 kg sand, then the mixture filled *Kachakupi* is placed at the exact centre and rest of the portion of the mud pot was filled with 10kg sand.
- Filling of sand should be done carefully to avoid entering of sand into the *Kachakupi*.
- Heating should be increased gradually.
- It should be allowed for self-cooling for the proper formation of the final product.
- *Kupi* should be taken out and broken carefully.
- Eyes and nose should be protected by wearing glasses and throughout the practical.

**Table No: 7 Showing results of *Silataalarasa***

| Results                          | Sample 1 | Sample 2 | Sample 3 |
|----------------------------------|----------|----------|----------|
| Quantity of drug used            | 51 g     | 53 g     | 54 g     |
| Total time taken for the process | 6 hours  | 6 hours  | 6 hours  |
| Amount obtained                  | 42 g     | 39 g     | 40 g     |
| Percentage of yield              | 82.35%   | 73.58%   | 74.14%   |
| Weight loss in percentage        | 17.64%   | 26.42%   | 25.86%   |

The above obtained drug is mixed with equal quantities of *trikatu choorna* and *Nirgundi moola choorna*.

**Table No: 8 Showing amount of final product obtained after mixing of other ingredients**

| Ingredients                          | Sample 1 | Sample 2 | Sample 3 |
|--------------------------------------|----------|----------|----------|
| Drug obtained after <i>Kupipakwa</i> | 42 g     | 39 g     | 40 g     |
| <i>Pippali</i>                       | 14 g     | 13 g     | 13.3 g   |
| <i>Maricha</i>                       | 14 g     | 13 g     | 13.3 g   |
| <i>Shunti</i>                        | 14 g     | 13 g     | 13.3 g   |
| <i>Nirgundimoola</i>                 | 42 g     | 39 g     | 40 g     |
| Total amount                         | 126 g    | 117 g    | 120 g    |

**Picture No: 1 showing various stages during *Silataalarasa* preparation**



**Picture No: 7 Showing *Kupi bhedhana***





Picture NO: 8 showing mixing of product with other herbal ingredients



Analytical study

The study has been divided into three parts

- 1. Physico chemical analysis
- 2. Instrumental analysis

Physico chemical analysis

Table No: 9 Showing organoleptic features of *Silataalarasa*.

| Sl No. | characteristics | Sample A       | Sample B       | Sample C       |
|--------|-----------------|----------------|----------------|----------------|
| 1      | Colour          | Dark brown     | Dark brown     | Dark brown     |
| 2      | Rupa            | Fine powder    | Fine powder    | Fine powder    |
| 3      | Odour           | Pungent        | Pungent        | Pungent        |
| 4      | Taste           | <i>Katu</i>    | <i>Katu</i>    | <i>Katu</i>    |
| 5      | Touch           | Very fine      | Very fine      | Very fine      |
| 6      | Sabda           | Not applicable | Not applicable | Not applicable |

Table No: 10 Showing physico chemical characteristics of *Silataalarasa*

| Sl No.                              | Characteristics            | Sample A      |
|-------------------------------------|----------------------------|---------------|
| <i>Bhasmapareeksha</i> <sup>9</sup> |                            |               |
| 1                                   | <i>Varna</i>               | Dark brown    |
| 2                                   | <i>Varitaratwa</i>         | Present       |
| 3                                   | <i>Rekhapurnatwa</i>       | Present       |
| 3                                   | <i>Mridu</i>               | Soft in touch |
| 4                                   | <i>Nischandratwa</i>       | Lustrous      |
| Modern parameters                   |                            |               |
| 1                                   | pH                         | 5.41          |
| 2                                   | LOD                        | 5.31%         |
| 3                                   | Total Ash                  | 5.04%         |
| 4                                   | Water soluble extractive   | 6.51%         |
| 5                                   | Alcohol soluble extractive | 13.97%        |
| 6                                   | Acid insoluble Ash         | 0.19%         |

Instrumental analysis

High-Performance Thin Layer Chromatography (HPTLC)

HPTLC analysis of *Silataalarasa* were carried out.

**Preparation of sample:**

1 g each of the above samples are taken, extracted with 10 ml *Methanol* separately and spotted as 10 microlitre. This was then applied on a precoated silica gel 60 F<sub>254</sub> on 10× 10 cm Aluminum sheet. The plate was then developed in *toluene: Ethyl acetate: Formic acid: Methanol* (7:5:1:0.5) in a twin trough chamber. The developed plates were visualized at 254, 366 nm, under white light. Rf and colour of the spots were recorded.

**Result:** at 254 nm total 8 peaks were identified and the total area of 98273.6 AU. At 366 nm, total 8 peaks were identified and an area of 17596.6 AU.

**XRD**

XRD analysis was done using the X-ray diffractometer by using X-ray wavelength, CuK alpha- 1.54056 Angstroms and with a power 45kV and 30mA. Sample was scanned from 11 degrees (2theta value).

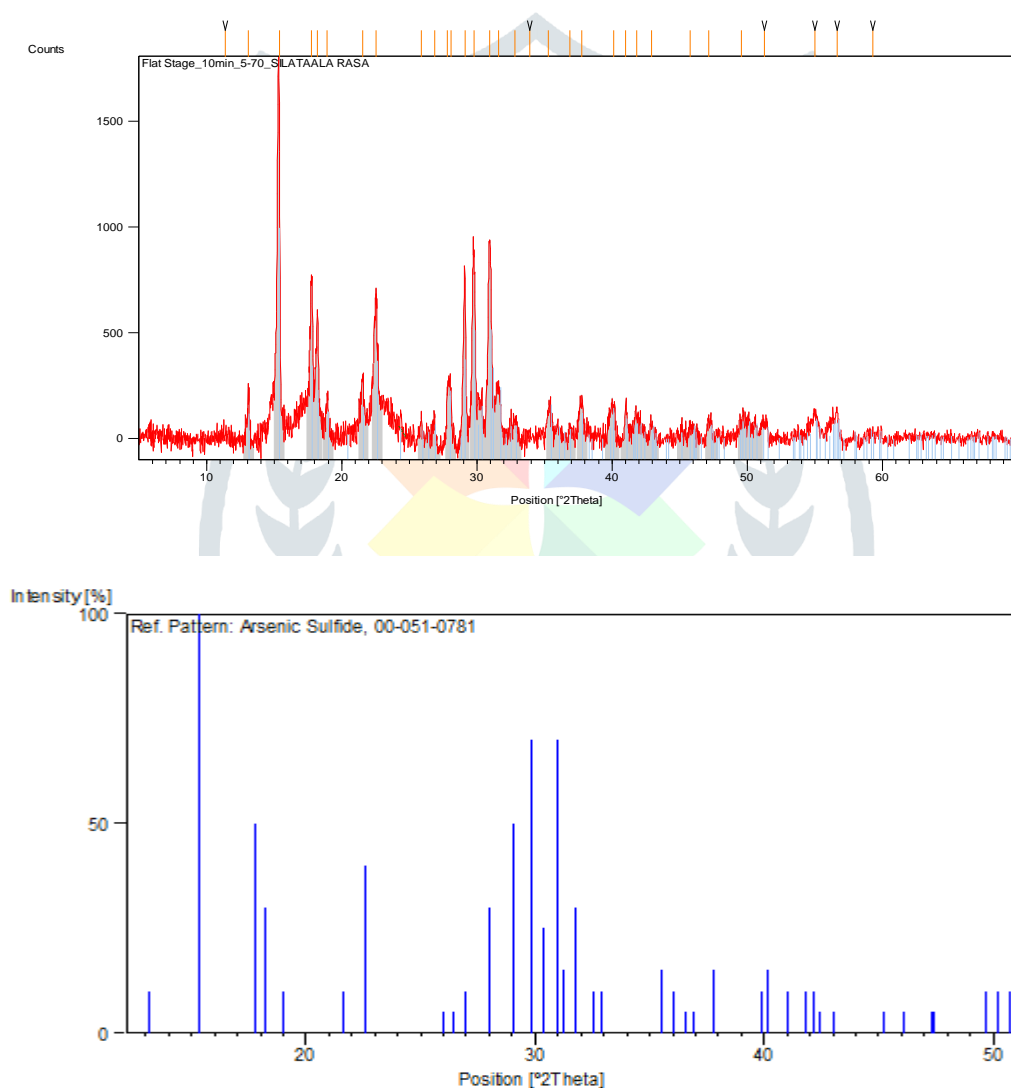
**Crystallographic parameters of the sample:**

Crystal system: monoclinic

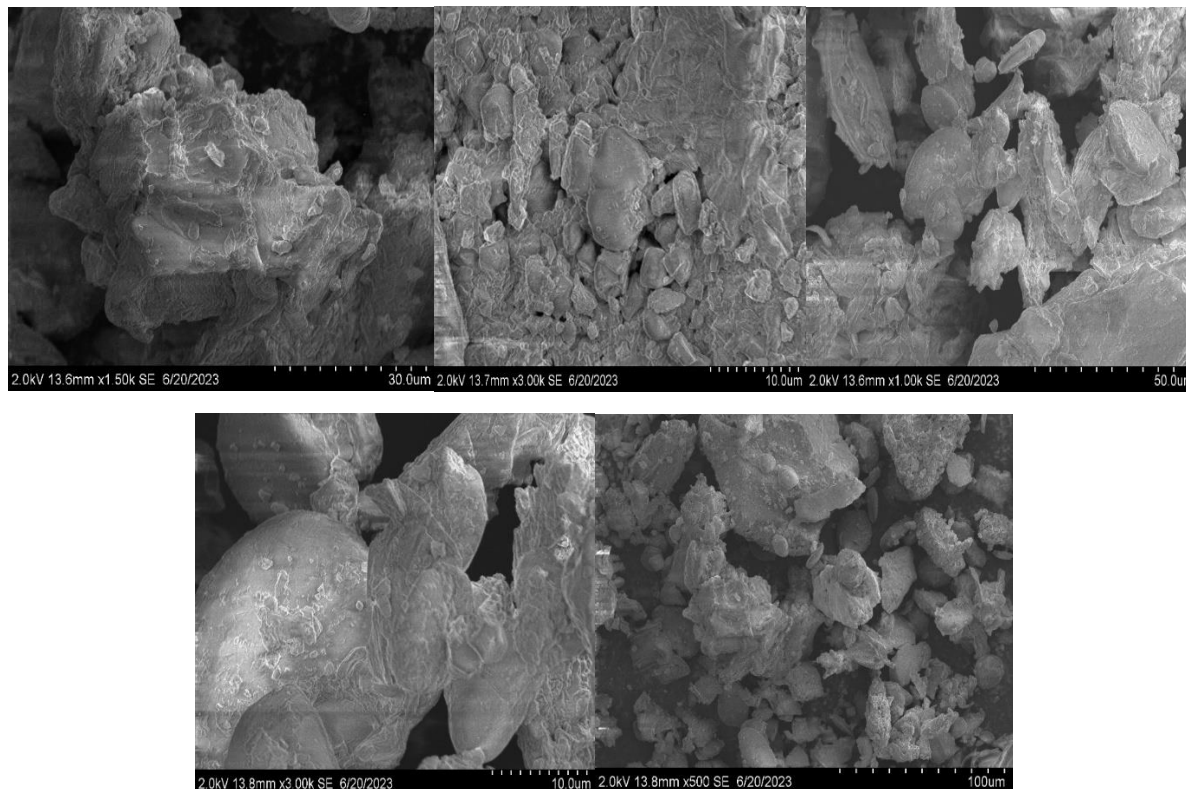
Space group: C2/c

Space group number: 15

Compound present in the sample: Arsenic Sulphide (As<sub>4</sub>S<sub>4</sub>)

**SEM (Scanning Electron Microscopy)**

Picture No: 2 showing SEM Analysis pictures



### EDS (Energy Dispersive X-ray Spectroscopy)

Processing option: All elements Analysed

Number of iterations: 4

**Table No: 11 Table showing EDS report of *Silataalarasa***

| Element found | Weight % | Atomic % |
|---------------|----------|----------|
| C             | 63.69    | 73.29    |
| O             | 28.14    | 24.31    |
| Si            | 0.18     | 0.09     |
| S             | 1.97     | 0.85     |
| K             | 1.78     | 0.63     |
| Ca            | 0.31     | 0.11     |
| As            | 3.94     | 0.73     |

### DISCUSSION AND CONCLUSION

*Silataalarasa* is a herbo-mineral formulation which is mainly indicated in *Swasa* and *Kasa roga*. Preparation of *Silataalarasa* was carried out according to the reference from *Brihat Rasa Raja Sundara*. The *Kupipakwa* method is utilized for preparing *Silataalarasa*. Although the *Kupipakwa Rasayana* preparation process is exhaustive and complicated, it holds significant importance due to its specific pharmaceutical procedures and remarkable clinical outcomes achieved with minimal therapeutic dosage. As per this reference, mixture obtained after *Bhavana* filled into the *Kupi* and subjected to heating in a *Valuka yantra* for 6 hours.

Organoleptic analysis shows all the samples were have dark brown colour powder with pungent order and taste. Also, very fine on touch. The samples satisfied *Vritaratwa*, *Rekhapoornatwa* and *Nishchandratwa*.

pH of the formulation is 5.41, suggest that the formulation is slightly acidic. Ash values are the criteria to judge the identity and purity of the drug. An Ash value of 5.04 indicates that approximately 5.04% of the formulation's weight consist of inorganic mineral matter (As). Loss on drying, this indicates the percentage of moisture content in the formulation that is lost when subject to drying conditions. LOD of this formulation is 5.31, suggests that the formulation contains a moderate level of moisture.

The HPTLC performed on the methanolic extract of *Silataalarasa* showed the presence of various constituents. The chromatogram scanned at 254 nm and 366 nm represent 8 peaks. The number of peaks indicates the presence of different chemical constituents in the sample. The cumulative area of all peaks is 98273.6 units. While comparing the standard Rf values, it suggests that this formulation may contain active compounds like *gingerol*, *piperine*, *vasicine*, *Glycyrolizin*, *eugenol* and *cineole*.



XRD was utilized to analyse the structural characteristics of the sample. The XRD result suggests that the analysed material is identified as *Arsenic Sulphide* ( $As_4S_4$ ). Crystalline in nature, monoclinic. Compared to oxide forms ( $As_2O_3$ )  $As_4S_4$  is less toxic.

SEM results reveals that the sample have an average particle size of  $10\mu m$  -  $100\mu m$ . sample was viewed repeatedly for 5 times under 1.50kx, 3.00kx, 1.00kx, 3.00kx, 500SE to identify the particle size of  $30\mu m$ ,  $10\mu m$ ,  $50\mu m$ ,  $30\mu m$ ,  $100\mu m$  respectively.

From the EDS analysis, it appears that carbon and oxygen are the dominant elements in terms of both weight and atomic percentages. These elements likely form the main structural components of the formulation. Other elements like *sulphur*, *potassium*, *calcium*, *silicon* and *arsenic* are present in smaller quantities. The presence of silicon might have come through the processing, because of the usage of *kachakupi*.

It is understood from the above studies that *Silataalarasa* is a compound with particle size in nanometre having carbon and oxygen as the major elements with sulphur, potassium and Arsenic as minor elements. Since it is indicated in *Swasa* and *Kasa*, the elements present in this formulation may have significant role in curing diseases.

## CONCLUSION

This study deals with the pharmaceutical and physicochemical evolution of *Silataalarasa*. It's made using the Kupipakwa method, which involves sustained gradual heating. This unique process increases the potency of the drug significantly over time. The final sample of *Silataalarasa* appeared fine, dark brown in colour, with a pungent smell and taste. Physico chemical analysis helps to generate a preliminary analytical profile for *Silataalarasa*.

## Conflicts of interest

I declare that I have no conflicts of interest.

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