



ISSN 2456-3110

Vol 5 · Issue 5

Sept-Oct 2020

Journal of  
**Ayurveda and Integrated  
Medical Sciences**

*www.jaims.in*

# JAIMS

An International Journal for Researches in Ayurveda and Allied Sciences



**Charaka**  
Publications

Indexed

# Pharmaceutico analytical study of *Shodhita Shilajatu*

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## ABSTRACT

**Background:** *Shilajatu* or *Adrija* is one of the *Maharasa*, which is considered as a wonderful medicine in Ayurveda. It is named as it comes out of the stones heated by the sun in summer in the form of thick blackish exudation having many shades. Since it contains stone, mud, wood, sand and various physical and metallic impurities, *Shodhana* (Purification) of *Shilajatu* is a mandatory procedure. It has been used as a prime ingredient in many formulations mainly for *Prameha*, *Sotha*, *Pandu Roga*, *Kshaya*, *Swasa*, *Pliha Vrudhi*, *Jwara*, *Agnimandya*, *Apasmara*, etc. **Objectives:** *Shodhana* of *Ashudha Shilajatu* and Physico chemical analysis of *Shodhita Shilajatu*. **Materials & Methods:** *Bhringaraja Swarasa* for *Shodhana* of *Shilajatu*. **Results:** It took 8 days for completion of *Shilajatu Shodhana*. XRD Analysis report indicates that the sample *Shilajatu* was Amorphous material. **Conclusion:** Total yield of *Shodhita Shilajatu* was 99.6%. The Sample of *Shilajatu* was found to be Amorphous material in XRD Analysis hence crystal structure was not identified.

**Key words:** *Shilajatu*, *Shodhita Shilajatu*, *Bhringaraja Swarasa*, *Suryatapi Method*, *XRD*.

## INTRODUCTION

*Shilajatu* is an important, known component of the Ayurvedic medicine given its characteristics as a *Rasayana*. According to *Rasaratna Samuchaya*, due to the heat of scorching sun rays in Summer, from the rocks consisting of metals like gold, silver, copper etc, in the form of a thick blackish exudation, *Shilajatu* oozes out.<sup>[1]</sup> *Shilajatu* mainly consists of humic substances like fulvic acid, humic acid, triterpenes, selenium, phospholipids and nearly 85 ionic minerals.

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Submission Date: 18/09/2020 Accepted Date: 22/10/2020

### Access this article online

Quick Response Code



Website: [www.jaims.in](http://www.jaims.in)

DOI: [10.21760/jaims.5.5.30](https://doi.org/10.21760/jaims.5.5.30)

It also contains non humic substances, which are low molecular weight compounds of marine fossil, plant and microbial origin, occurring in and around *Shilajatu* bearing rocks..It mainly contains fulvic acid which is an important mineral. It is a richest source of ionic minerals and other elements which are commonly depleted in our soil today.<sup>[2]</sup> It is found in Himalayan regions, Kashmir, Bhutan and Tibet.<sup>[3]</sup> *Shilajatu* has been classified in to 4 types , which are *Swarna*, *Rajata*, *Tamra* and *Loha*.<sup>[4]</sup> *Shilajatu* needs proper purification processes to remove the physical impurities and to enhance the therapeutic efficiency of it. If it is consumed without proper *Shodhana*, it causes *Daha*, *Murcha*, *Bhrama*, *Raktapitta*, *Sosha* and *Agnimandya*.<sup>[5]</sup> There are various *Shodhana Drava Dravyas* like *Godugda*,<sup>[6]</sup> *Gomutra*,<sup>[7]</sup> *Triphala Kwatha*,<sup>[6],[7]</sup> *Bhringaraja Swarasa*<sup>[6],[8]</sup> mentioned for *Shilajatu Shodhana*. *Shilajatu* can be purified either by *Agnitapi* method or *Suryatapi* method. *Acharya Charaka* in 1st Century A.D quoted that "There is hardly any curable disease which cannot be controlled or cured with the aid of' *Shilajatu*.<sup>[9]</sup> Traditional uses primarily focus on *Prameha* and diseases of the

urinary tract, but also include *Sotha, Gulma, Kshaya, Apasmara, Unmada, Pleeha, Krimi, Vatarakta, Arsas, Pandu*, etc.<sup>[10],[11]</sup>

## MATERIALS AND METHODS

### Pharmaceutical study - Shodhana of Ashudha Shilajatu

Confirmatory test for *Grahya Shilajatu* :

- When *Shilajatu* is put on fire, it must burn without producing any smoke and should take the shape of *Linga*.<sup>[12],[13]</sup>
- When dropped into water, it should spread like a thread to the bottom without getting dissolved.<sup>[13],[14]</sup>
- It should have black colour and emit *Gomutra Gandha*.
- It should have *Katu* and *Tikta Rasa*.<sup>[12]</sup>

### Materials

- Ashuddha Shilajatu* : 250gms, collected from Dorle Ayurvedic, Pune.
- Bhringaraja Swarasa* : 1 L

### 1) Method of Shodhana

- 250 grams of *Ashuddha Shilajatu* was taken and made into Coarse powder by using *Khalwa Yantra*.
- Bhringaraja Swarasa* was prepared and filtered.
- This mixture was kept on stove, heated on *Mandagni* with constant stirring the mixture.
- When mixture attained syrup consistency, the vessel was taken off from the stove and kept it under Sunlight.
- The creamy layer which appeared on the surface was gently removed and poured on a ghee smeared plate and kept it under the sunlight again for drying.
- This was continued everyday till the *Shilajatu* dried off completely.
- The process was carried out for 8 days.

- After complete drying, *Shilajatu* was removed from the plate powdered and stored in container.

### 2) Physico chemical analysis of Shodhita Shilajatu

#### Materials

*Shodhita Shilajatu Churna* (for Organoleptic, Physical constituents, Phyto chemical Analysis, SEM EDX and XRD Analysis)

#### Method

##### A. Organoleptic characters of *Shilajatu*<sup>[13]</sup>

Organoleptic characters of *Shodhita Shilajatu Churna* were tested with sense organs.

##### B. Physical Constituents

##### 1. Determination of Moisture Content<sup>[15]</sup>

5 gm of dried sample of *Shilajatu Choorna* (W) was taken in a clean and dry petridish. Weight of the petridish with sample was noted (W<sub>1</sub>). It was kept in a hot air oven for an hour. Later it is taken out and weighed. Again the petridish was placed in the Hot air oven for an hour, taken out and weighed ( at 105°C). This procedure was repeated until the constant weight of petridish containing the powder sample was obtained (W<sub>2</sub>). The Moisture content was calculated as follows,

% of Moisture content = Difference of weight / Weight of the sample \* 100

% of Moisture content =  $W_1 - W_2 / W * 100$

##### 2. Determination of Ash Value

Weight of an empty clean and dry crucible was noted (W). 5 gm of air dried *Shilajatu Churna* was taken and transferred to it. The crucible along with *Shilajatu Churna* was weighed(W<sub>1</sub>).Then it was subjected to incineration in an electric burner at a temperature not exceeding 450°C. till carbon free ash was obtained. The crucible was then taken out, cooled and weighed (W<sub>2</sub>). Ash value is calculated as follows,

Total Ash value = W<sub>2</sub> – W

% of Total Ash =  $W_2 - W / \text{Wt. of sample} * 100$

### 3. Determination of Acid Insoluble Ash

Total ash obtained above was treated with 100 ml of 6N dilute Hydrochloric acid and boiled. Insoluble matter was collected on ashless filter paper Whatman No. 42. The residue was repeatedly washed with hot water, dried well and ignited in electric burner cooled and weighed. Percentage of acid insoluble ash was calculated as follows,

Weight of filter paper with ash - weight of filter paper / Weight of ash \* 100

### 4. Determination of Water Insoluble Ash

5 gms of air dried *Shilajatu Churna* was taken in a silica crucible. It was kept in electric burner for incineration till carbon free ash was obtained. The ash was taken out, cooled and boiled with 25 ml distilled water for 5 minutes and cooled. It was then filtered by using ash less filter paper Whatman No. 42. Then washed with hot water for few times. Dried well and filter paper was ignited along with the insoluble matter in an electric burner in a crucible. The ash is taken, cooled and weighed. The Water insoluble ash was calculated as follows,

Weight of filter paper with ash - weight of filter paper / Weight of ash \* 100

### 5. Determination of Specific Gravity

Empty Picnometer was weighed ( $W_1$ ). It was filled with distilled water and weighed ( $W_2$ ). Picnometer was filled with 1% of sample solution (i.e., 100 ml of distilled water + 1 gm of *Shilajatu Churna*) and weighed ( $W_3$ ). The specific gravity of sample was calculated as follows,

Specific gravity of the sample =  $W_3 - W_1 / W_2 - W_1$

### 6. Determination of pH

1% of test drug solution (100 ml of distilled water + 1 gm of *Shilajatu Churna*) was prepared. pH meter was taken and tip of the electrode was dipped in to the solution and kept undisturbed for 1-2 min. and the value was recorded.

### 7. Percentage of Solubility

A china dish was taken and weighed ( $W_1$ ). 5gm of fine powder of *Shilajatu* ( $W_2$ ) was mixed with 100 ml of

distilled water and stirred well. It was kept for 24 hours and filtered through Whatman filter paper no. 42. The filtrate was poured in China dish and then China dish was kept on water bath for drying. The filter paper along with residue was dried and weighed. The China dish was weighed along with sample ( $W_3$ ). Percentage of solubility was calculated as follows,

% of solubility of *Shodhita Shilajatu* Sample =  $W_3 - W_1 / W \times 100$  (in DW)

## C. Phyto-Chemical Analysis<sup>[15]</sup>

### 1. Detection of Alkaloids

Test drug sample solution + Wagner's reagent (KI solution). Reddish brown colored precipitate was not observed in test drug sample.

### 2. Detection of Carbohydrates

Benedict's test: (test for reducing sugars)

2ml of Benedict's reagent and 1 ml of test drug sample solution was heated in a test tube. Brick red precipitate was not observed.

### 3. Detection of Flavanoids

Sodium Hydroxide solution test: Test drug sample Solution + 10% NaOH solution. Yellow colour was observed in sample, became colourless on the addition of few drops of dilute acid..

### 4. Detection of Tannins

Test drug sample solution +  $FeCl_3$  ( 2-3 drops). Blue colour was observed in test tube.

### 5. Detection of Triterpenoids

Salkowski test : Test drug dry extract (2 mg) + concentrated  $H_2SO_4$  (few drops) + Chloroform( 1 ml) . Reddish brown colour was observed.

### 6. Detection of Steroids

Salkowski test : Test drug sample Solution + concentrated  $H_2SO_4$  + Chloroform. Blood red colour was observed in the test drug sample

## 7. Detection of Proteins

Million's Biuret test: Test drug sample Solution + 2ml 10% NaOH solution + 2ml 1% CuSO<sub>4</sub>. Purplish blue colour was observed in sample.

## 8. Detection of Saponins : ( Foam index test )

1 ml extract of test drug solution + 20ml distilled water, and shaken for 15 minutes. Foam was observed in sample.

## 9. SEM EDX - (Scanning Electron Microscopy) & (Energy Dispersive X-Ray Spectroscopy)

SEM is a powerful and mature technique in the examination of materials, widely used in Metallurgy, Geology, Biology and Medicine. It can obtain high magnification images with a good depth of field. It can also analyze individual crystals or other features.

When used in conjunction with the closely-related technique of energy-dispersive X-ray microanalysis (EDX, EDS, EDAX), the composition of individual crystals or features can be determined.

*Shodhita Shilajatu* sample was subjected to SEM EDX, but the sample got burnt due to high voltage, thereby couldn't perform the test.

## 10. XRD (X-ray Diffraction)

It is a non destructive technique that provides detailed information about the crystallographic structure, chemical composition and physical properties of materials. The *Shodhita Shilajatu* sample was subjected to XRD Analysis, the results are shown in graph no. 1.

## OBSERVATIONS AND RESULTS

### 1) Pharmaceutical study

1. *Bhrungaraja Swarasa* was dark green in colour which stained the hands.
2. It took around 2 hours to obtain *Swarasa*.
3. Physical impurities like stones and sand was obtained during filtration.
4. The colour of supernatant layer on *Shilajatu* containing vessel was Blackish brown.

5. The final product of *Shilajatu* was pitch black in colour and it was in the form of flakes.

**Table 1: Showing the quantity of *Bhrungaraja Swarasa*, time taken & weight of *Shilajatu* in *Shodhana* process.**

Quantity of <i>Shilajatu</i> before <i>Shodhana</i>	Quantity of <i>Bhrungaraja Swarasa</i>	Quantity of <i>Shilajatu</i> after <i>Shodhana</i>	Time taken to complete the process	Days taken to obtain <i>Suryatapi Shilajatu</i>
250gms	1 L	249 gms	3 hrs	8 days

**Yield = 99.6 %**

**Loss = 0.4 %**

### Precautions

- The *Suryatapi* method of *Shodhana* of *Shilajatu* should be carried out preferably in Summer as it needs strong sun rays.
- The vessel should be kept in an open space like Terrace so that it gets sufficient and unobstructed sunlight.
- In the evening, after drying in the Sun, the vessel should be kept indoors by properly closing it with a lid so that no flies or dust enters in.
- The creamy thick layer should be collected everyday and carefully transferred in to a wide ghee smeared glass plate so that its easy to collect it later.

### 2) Physico chemical analysis

**Table 2: Showing Organoleptic characteristics of *Shodhita Shilajatu***

SN	Organoleptic Characters	<i>Shodhita Shilajatu</i>
1.	Colour	Blackish brown
2.	Odour	<i>Gomutra Gandhi</i>
3.	Taste	Astringent
4.	Touch	Smooth
5.	Appearance	Lump

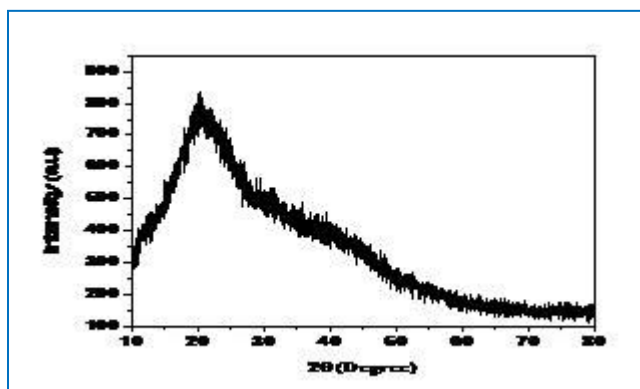


**Table 3: Showing Physical constituents of Shodhita Shilajatu.**

SN	Physical constituents	Results
1.	Moisture content	1 %
2.	Ash value	19.2 %
3.	Acid insoluble Ash	1 %
4.	Water insoluble Ash	13.6 %
5.	Specific gravity	1.009
6.	pH	6.5
7.	% of Solubility	74% (in DW)

**Table 3: Showing Phyto chemical constituents of Shodhita Shilajatu.**

SN	Phyto chemical constituents	Result
1.	Alkaloids	-ve
2.	Carbohydrates	-ve
3.	Flavanoids	+ve
4.	Tannins	+ve
5.	Triterpenoids	+ve
6.	Steroids	+ve
7.	Proteins	+ve
8.	Saponins	+ve

**Graph 1: Showing the analysis of XRD report**

The sample is Amorphous material, so crystalline structure is not found.

## DISCUSSION

*Shilajatu* is considered as the best therapeutic and *Rasayana* agent which makes the body strong, free from all the diseases and grant longevity to the human body. It is composed of humus and organic plant material that has been compressed by layers of rock mixed with microbial metabolites. *Shilajatu* has been in extensive use in the preparations of a number of medicines. It is mainly used in the treatment of *Jwara*, *Rajyakshma*, *Kshaya*, *Prameha*, *Gulma*, *Pleeha*, *Asmari*, *Muthrakrichra*, *Swasa*, *Pandu*, *Apasmara*, *Unmada*, etc.

*Shilajatu Shodhana* was carried out as per the reference of *Rasa Ratna Samucchaya* - 2/ 110-111 and *Suryatapi* method was adopted. *Bhrungaraja Swarasa* was used as *Shodhana Dravya*. The herb *Bhrungaraja* is considered to be a *Rasayana* in Ayurveda and works wonderfully on *Tridosha* and maintains their balance which might be the probable reason for using it for *Shodhana* since it augments the *Rasayana* effect of *Shilatu*. *Shilajatu* was powdered in coarse form and subjected to heat after adding *Bhrungaraja Swarasa* to it and filtered to remove physical impurities. The heating was continued till the liquid attained syrup consistency. Later the vessel was placed on terrace under sunlight. The brownish creamy layer which appeared on the surface was gently removed everyday and kept it for shadow drying. The process took 8 days to finish and obtain *Shodhita Shilajatu* and the final product was pitch black in colour. Total yield of *Shodhita Shilajatu* was 99.6%.

The sample has shown organoleptic characters like blackish brown colour, *Gomutra Gandhi* smell, astringent in taste, smooth in touch and lump like appearance. The sample was subjected to Physical constituents Analysis. Moisture content was 1%, Ash value was 19.2%, Acid Insoluble ash was 1%, Water insoluble ash was 13.6%, Specific gravity was 1.009%, pH was 6.5% and Percentage of solubility was 74% in distilled water.

The Phyto chemical constituents like Flavanoids, Tannins, Triterpenoids, Steroids, Proteins, Saponins were present and Alkaloids and Carbohydrates were absent in the sample.

XRD report indicates that the sample is amorphous material, which means it doesn't have a detectable crystal structure. It points towards the hypothesis that *Shilajatu* is produced by the decomposition of plant *Asphaltum panjabinum*.

## CONCLUSION

*Bhrungarja Swarasa* was used as the *Shodhana Dravya* for *Shilajatu*. *Suryatapi* method of *Shodhana* was adopted which took 8 days for completion as the *Shodhana* was done during Summer season. Total yield of *Shilajatu* was 99.6%. XRD analysis indicated that *Shilajatu* sample was amorphous, meaning which doesn't have a detectable crystal structure.

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**How to cite this article:** Arya Elias, Pramod C. Baragi, Kashinath Hadimur, K. A. Patil, Khazi Rahimbi. Pharmaceutico analytical study of Shodhita Shilajatu. J Ayurveda Integr Med Sci 2020;5:214-219. <http://dx.doi.org/10.21760/jaims.5.5.30>

**Source of Support:** Nil, **Conflict of Interest:** None declared.

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