



EUROPEAN JOURNAL OF BIOMEDICAL AND PHARMACEUTICAL SCIENCES

<http://www.ejbps.com>

ISSN 2349-8870

Coden USA: EJBPO

Volume: 13

Issue: 4

Year: 2026

PHARMACEUTICAL AND ANALYTICAL STUDY OF GOMUTRA SHILAJATHU

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DOI: <https://doi.org/10.5281/zenodo.19508938>



How to cite this Article: *Dr. Anjana P. (2026). Pharmaceutical and Analytical Study of Gomutra Shilajathu. European Journal of Biomedical and Pharmaceutical Sciences, 13(4), 519-525.

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Article Received on 15/03/2026

Article Revised on 05/04/2026

Article Published on 10/04/2026

ABSTRACT

Shilajatu is one of the Rasadravya (herbomineral drug) mentioned in the classical texts. Shilajatu is obtained from the mountain rocks in the form of exudate which oozes out in summer season. As per Charaka Samhita metals like gold and others are present in the rocks which receive the sun rays and secrete the exudate which is known as shilajatu. Ayurveda mentions it is as a wonderful medicine. Sodhita Shilajatu after Bhavana can cure many diseases. Shilajatu mainly divided in to 2 types, among them Gomutra Gandhi Shilajatu is considered to be the best one. Gomutra Shilajatu can be purified by Agni thapi or Sooryathapi method using Triphala Kwatha. Rasa Taragini mentioned special method of preparation of Triphala Kwatha for Gomutra Shilajatu Sodhana. Among them Sooryathapi method is highly recommended because of more yield compared to other method. In the present study, Sodhitha Shilajathu were analysed with Classical as well as modern parameters.

KEYWORDS: Gomutra Shilajathu, Soorya Thapi Method, Pharmaceutical study, Analytical study, Triphala Kwatha.

INTRODUCTION

Shilajatu is an exudation from rock found during hot sunny days. Though it may be occurring in many parts of the world but India was the first to highlight its tremendous therapeutic value for many centuries BC (era of ancient Indian physician Charaka & Susruta). Charaka

says “there is hardly any curable disease which cannot be controlled or cured with the aid of Shilajatu.”^[1] Shilajatu is included under Maha Rasa group, by almost all the Rasa Sastra classics, but the Nighantus placed it in different groups according to their classification. Rasa Targini included Shilajathu under Misralohadi Varga.

- Vernacular Names^[2]: Different vernacular names of Shilajatu are as follows:

Arabic	– Hajar – ul – Musa	Marathi	– Shilajita
Bengali	– Shilajita	Malayalam	– Kanmadam
Dravida	– Urugum	Nepali	– Kalo Shilajita
English	– Mineral Pitch	Parsi	– Momiya Phacyral Yahud
Gujarati	– Shilajita	Sanskrit	– Shilajatu
Hindi	– Shilajita	Unani	– Momiye or Shilajita
Latin	– Asphaltum punjabinum		

Shilajatu is an exudate oozing out from the rocks due to the heat of scorching sun rays in hot weather^[3] in lower Himalayas, Vindhya and also mountains of Bhutan, Tibet and Nepal. In India it is available from Kashmir and Punjab. Different types of Shilajatu are explained in various texts as far as the number is concerned. But the

basic criterion remains same that is the variety depends up on the rock, from where it exudes.

Table No. 1: Shilajatu Classifications in Samhitas.

Varieties	C.S	Su.S	A.H
Swarna	+	+	+
Rajatha	+	+	+
Tamra	+	+	+
Lohaja	+	+	+
Nagajam	--	+	+
Vangajam	--	+	+

Table No. 2: Shilajatu classification in Rasa Sastra.

Text	Varieties
R.R.S	I. Gomutra Gandhi and Karpura Gandhi II. Swarnaja, Rajathaja and Tamraja
R.T	Swarnaja, Rajatham, Tamram, Lohajam
RSS/ RJN	Girijam and Usharaka
Rasarnava	Pathitha and Apathitha
A.P/ RPS	Girisambhavam and Ksharabhoomija

Gomutra Gandhi Shilajatu is again subdivided in to Sasatwa and Nihsatva.^[4] Among the two varieties of Shilajathu, Gomutra Shilajathu is considered to be the superior one. Shilajatu, being an exudate from the rocks, the impurities acquired in contact with metals, soil, and water should be removed to make it therapeutically eligible. Basically 2 methods are explained for the purification of Shilajatu in classical text books. **Sooryatapi method:** This is the method of purification of Shilajatu by exposing to sunlight. Shilajatu soaked either in milk or some Kashaya or some Swarasa in an iron vessel for required period under shade. After exposure to the sunlight for a period of 3 to 5 days, a layer of pure Shilajatu will be formed at the centre of the iron vessel. It should be collected and the rest of the liquid should be exposed to the sunlight again. **Agnitapi method:** Shilajatu is purified by heating in liquid medium. It is placed on a cloth tied to the mouth of the vessel. Shilajatu purified by this method is said to be inferior to the Sooryatapi Sodhita Shilajatu. Because of the exposure to heating, will lead to loss of some volatile principles and chemical constituents.^[5]

AIM

- Pharmaceutical purification of Gomutra Shilajathu
- Analytical study of Asodhitha and Sodhitha Gomutra Shilajathu by Classical and Modern Parameters.

PHARMACEUTICAL STUDY

Pharmaceutical study mainly includes collection of crude drugs and pharmaceutical processing like Shodhana. By this impurities are separated from the substances by various processing with specific herbal drugs. Shodhana is a process of purification and detoxification by which physical, chemical blemishes and toxic materials are eliminated and substances can be subjected for further processing.

Shilajatu is a blackish brown exudation of variable consistency found in the serene surroundings of

Himalayas. It is composed of humus and organic plant material that has been compressed by layers of rock mixed with microbial metabolites.

SHILAJATU SODHANA

- **Procurement**
- Crude genuine samples of Gomutra Gandhi shilajatu was collected from Himachal Pradesh and Bombay through a G.M.P certified Factory Udayamperoor, Ernakulam, Kerala.
- **Physical characteristics of Raw Gomutra Shilajatu:**
Colour – Blackish brown
Smell – Intense smell of Cows urine
Texture – Black stony mass
Taste – Characteristic

- **Test for genuinity**

Obtained sample of Raw Gomutra Shilajatu was compared with the genuine sample at Q&A department of Kottakkal Arya Vaidyasala Malappuram and was proved to be genuine.

- **Classical Shilajatu Pareeksha^[6]**

1. When the raw sample was put into fire, it burns without any smoke.
2. When it was put in to fire, it attained a shape of a Lingam.
3. When it was dropped into water with the help of a spatula, it sank down without dissolving in water.
4. Had intense odour of Gomutra.

- **SHILAJATU SODHANA^[7]**

- Materials required:

Raw Gomutra Shilajatu – 1kg

Triphala Kwatha – 500ml

Hot water – 2L

Preparation of Triphala Kwatha^[8]

- Ingredients needed:

Coarse powder of Triphala – 1kg

Water - 8 L

- Procedure: The coarse powder of triphala was taken in a decoction vessel in equal quantity to that of raw Shilajatu. It is added with eight times of water and cooked over mild fire. When the liquid was reduced to quarter, the liquid was filtered and used as Triphala Kwatha for Shilajatu Sodhana.

Detailed method of purification of Shilajatu

- Materials and methods

Wide mouthed vessels, trays, spoon, cloth, stove etc.

- **Procedure**

- ❖ In a wide mouthed vessel, one part of raw shilajatu choorna was taken. It was added with two parts of hot water and half part of Triphala Kwatha.
- ❖ The mixture was stirred well to dissolve the Shilajatu completely.

- ❖ The vessel is kept undisturbed under sunlight for 3 hours.
- ❖ Later the mixture was macerated well and filtered through a strong cloth in to a second wide mouthed vessel.
- ❖ The second vessel containing the blackish solution of Shilajatu was placed under hot sun.
- ❖ When a blackish creamy layer surfaced over the liquid, it was carefully skimmed off in to the third vessel containing required quantity of Triphala kwatha.
- ❖ When the blackish creamy layer resurfaced in the third vessel, it was again skimmed off to the fourth vessel containing required quantity of Kashaya.
- ❖ The process of placing the vessel, containg creamy layer of Shilajatu and Kashaya under hot sun and skimming off the surfaced creamy layer in to another vessel was continued until; no more creamy layer appeared over the surface.
- ❖ The last vessel containg the mixture of Shilajatu and Kashaya was subjected to mild fire.
- ❖ The Kashaya got reduced and thick black paste of Shilajatu was obtained in the vessel.
- ❖ Finally the thick black paste of Shilajatu is skimmed off in to a clean stainless steel tray.
- ❖ It was dried under the sun and preserved in wide mouthed glass bottles.

▪ OBSERVATION

- ❖ At first Shilajatu, which was in black stony mass, was crushed in to small pieces. After crushing the Gomutra Gandha was more intensive.
- ❖ It took about 2 hours for the complete mixing of Shilajatu Choorna in Triphala Kwatha.
- ❖ The physical impurities like sand and small stone particles remained undissolved in the Kashaya.
- ❖ No specific colour change was observed when the Shilajatu mixed Kashaya was placed under the sun for 3 hours.

• Results

Table No 3. Result of Shilajatu Sodhana.

Batch	Quantity	Triphala Kwatha	Wt. of Sudha Shilajatu	Loss
I	1kg	500ml	850gm	150gm
II	4kg	2L	3.452kg	548gm
III	4kg	2L	3.779kg	221gm
IV	1kg	500ml	795gm	205gm

Total quantity of Raw Shilajatu – 10kg

Total quantity of Sudha shilajatu – 8.876kg

Total loss – 1.124kg



Fig: 1. Asudha Gomutra Shilajatu.



Fig: 2 Shilajatu with Triphala Kashaya.



Fig: 3. Shilajatu with Triphala Kashaya placed under sun.



Fig: 4. Solution placed under sunlight after filtering.



Fig: 5. Layer of sudha Shilajatu surfacing over the liquid.



Fig: 6. Reduction of final solution by mild heating.



Fig: 7. Layer of Sudha Shilajatu.

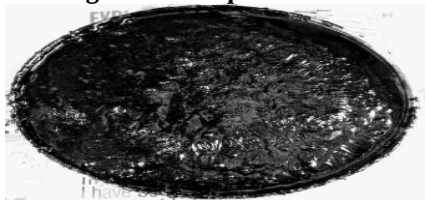


Fig: 8 Sudha Shilajatu obtained after reduction.



Fig: 9. Sudha Shilajatu after proper drying.

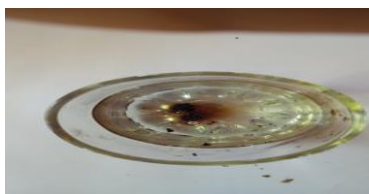


Fig: 10. Shilajatu Pareeksha in water.



Fig: 11. Lingakaram of Shilajatu.

ANALYTICAL STUDY PLAN OF THE STUDY

1. Samples

The samples analysed were as follows.

Raw Gomutra Shilajatu

Sudha Gomutra Shilajatu

2. Study Setting

- Quality and Assurance department, Arya Vaidya Sala, Kottakkal.
- Sophisticated Test and Instrumentation Centre (STIC), CUSAT, Kochi.
- Care Keralam Ltd, KINFRA Park, Koratty, Thrissur.

3. Methods

The samples were subjected to analytical study for the following parameters.

Raw Gomutra Shilajatu

- Classical Shilajatu Pareeksha
- Ash value
- Acid insoluble ash
- Water soluble extractive
- Alcohol soluble extractive.

Sudha Gomutra Shilajatu

- Loss on drying (moisture content)
- Ash value
- Acid insoluble ash
- Water insoluble ash
- Water insoluble extractive
- Alcohol insoluble extractive
- HPTLC fingerprint

OBSERVATIONS AND RESULTS

• Organoleptic evaluation

Table No. 4: Organoleptic Characters.

Parameter	Raw Gomutra Shilajatu	Sudha Gomutra Shilajatu
Colour	Blackish brown	Blackish brown
Odour	intense smell of Cows urine	Intense smell of cows urine
Taste	Characteristic	specific
Touch	Black stony mass	Thick black paste

Table No. 5: Observations of classical parameters of Shilajatu.

Parameter	Raw Gomutra Shilajatu	Sudha Gomutra Shilajatu
Salilae avaleenam	+	+
Adhoomakam	+	+
Lingakaram	+	+
Gomutra Gandha	+	++

• Analysis of modern parameters

1. Raw Gomutra Shilajatu

- Ash value- 25.54%w/w
- Acid insoluble ash – 0.52%w/w
- Water soluble extractive - 88.01%w/w
- Alcohol soluble extractive- 45.34%w/w
- LOD (moisture content) – 7.56%w/w

2. Sudha Gomutra Shilajatu

- Ash- 22.06%w/w
- Acid insoluble ash – 0.86%w/w
- Water insoluble extractive- 38.41%w/w
- Alcohol insoluble extractive- 58.10 %w/w

Result of High performance thin layer chromatography analysis

- **Sample details:** Sudha Gomutra Shilajatu
- **Test solution:** 01.1g each of above samples was weighed extracted with 10ml Methanol separately and spotted as 10 microlitre.
- **Stationary phase:** Merk, 1.05554.0007, TLC Silica gel 60 F₂₅₄, 10*10 cm Aluminium sheet.
- **Mobile phase:** Toluene: Ethyl acetate: Formic acid: Methanol (7:5:1:0:5)
- **Development:** CAMAG 10*10 cm Twin trough chamber.
- **HPTLC Instrumentation:** CAMAG Linomat 5, CAMAG TLC Scanner 3, CAMAG Reprostar 3.
- **Derivatization:** Iodine vapour.

▪ Results

Table No. 6: R_f value and % area of Sudha Gomutra Shilajatu sample at 254nm.

Peak No	R _f Value	Area (AU)	% Area (AU)
1	0.02	1969.1	2.19
2	0.05	361.4	0.40
3	0.07	493.0	0.55
4	0.12	319.8	0.36
5	0.19	1848.1	2.06
6	0.24	3861.8	4.30
7	0.26	2715.4	3.02
8	0.31	4748.8	5.29
9	0.34	21438.7	23.86
10	0.52	46984.1	52.31
11	0.70	5081.1	5.66

Total peak No: 11. Total area: 89821.3 (ASU).

Table No. 7. R_f Value and % area of Sudha Gomutra Shilajatu sample at 366nm.

Peak No	R _f Value	Area (AU)	% Area (AU)
1	0.22	182.1	3.03
2	0.27	731.3	12.17
3	0.37	2253.5	37.51
4	0.42	1342.2	22.35
5	0.47	1498.5	24.94

Total peak No: 05, Total area: 6007.6 (AU).

Table No. 8: R_f value and % area of Gulika sample at 254nm.

Peak No	R _f Value	Area (AU)	% Area (AU)
1	0.09	235.1	0.48
2	0.13	1264.9	2.59
3	0.18	13733.1	28.09
4	0.22	6173.0	12.63
5	0.28	4541.6	9.29
6	0.32	1367.1	2.80
7	0.35	4955.3	10.14
8	0.41	1420.6	2.91
9	0.49	8947.0	18.30
10	0.60	422.9	0.87
11	0.69	5829.0	11.90

Total peak No: 11

Total areas: 48889.6 (AU).

Table No. 9: R_f value and % area of Gulika sample at 366nm.

Peak No	R _f value	Area (AU)	% Area (AU)
1	0.09	1147.2	1.87
2	0.17	3909.5	49.68
3	0.28	1307.1	16.61
4	0.30	767.0	9.75
5	0.37	794.8	10.10
6	0.48	606.2	7.70
7	0.64	337.8	4.29

Total peak No. 07: Total area: 8869.6 (AU).

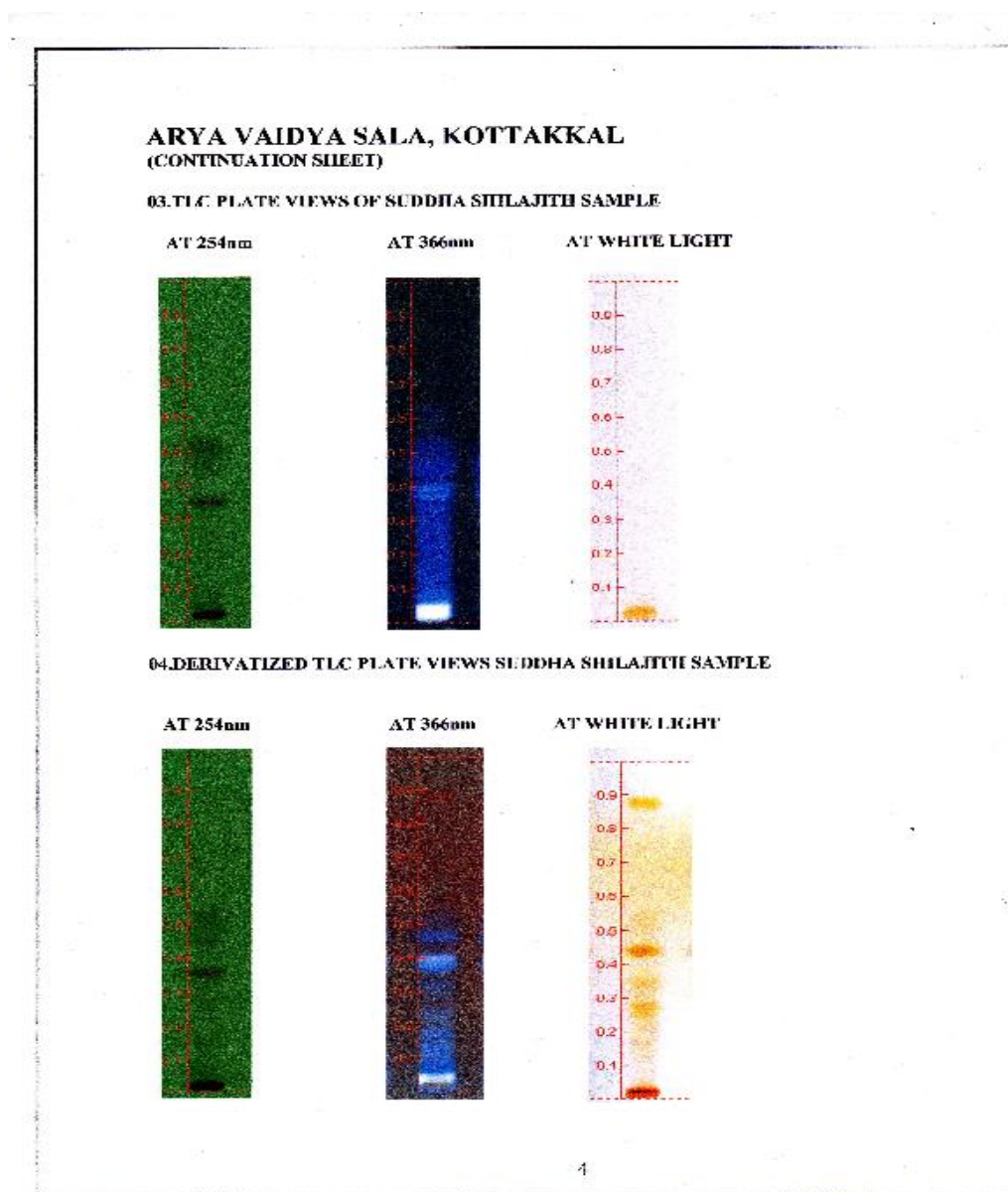


Fig no. 12: HPTLC pattern.

DISCUSSION

- Impure Shilajatu if taken internally may produce Daha, Pitha prakopa, Raktavikara, Moorcha, Agnimandhya and Malabadhata. Hence it is

necessary to purify the Shilajatu before medicinal use. Triphala Kwatha was used as the purificatory media, where the amount of Triphala kwatha was taken double to that of Shilajatu. But the amount

seemed insufficient for dissolving the Shilajatu and it was found that the solution became very thick. Hence equal amount of hot water was added. Each time the surfaced layer of Shilajatu was completely removed and added with fresh Triphala Kwatha. When no more creamy layer surfaced over the liquid, the remaining solution was subjected to mild fire to extract the remaining Shilajatu. Reason for using Triphala Kwatha may be because of its properties of Tridosasamana (especially Kapha) that is why it is added in Shilajatu and it augments the effect in reducing the Kapha dosha. This may be due to the fact that solid portion of Triphala kwatha increases the weight of Shilajatu.

Reason for selection Sooryatapi method: Sooryatapi Shilajatu is considered as superior one, as the volatile oils or contents present in it do not get destroyed.

The Raw Shilajatu and Sudha Shilajatu were subjected to classical Shilajatu Pareekshas. The Lingakara Lakshana was seen and this may be because of the presence of fulvic acid in the sample. Acid insoluble ash of Asudha Shilajatu and Sudha Shilajatu were 0.52% and 0.86% respectively and result signifies that the considerable amount of Sudha Shilajatu is soluble in acidic media of stomach. Total ash value of Asudha Shilajatu and Sudha Shilajatu were 25.54% and 22.06% respectively. So the Sudha Shilajatu is more stable and bio available than raw Shilajatu. Raw Shilajatu and Sudha Shilajatu possessed 7.56% and 14.31% loss on drying at 110°C. Hence it can be stated that Raw Shilajatu has least amount of moisture content, and Sudha Shilajatu is having high moisture content which may be because of the process followed in its purification.

HPTLC pattern of Sudha Shilajatu was compared with the standard pattern of Shilajatu and showed nearly same peaks, indicating the genuinity of the sample.

CONCLUSION

Shilajatu is a humus rich blackish –brown substance, which is having large spectrum of indications and serves as a potent tonic. Its source of origin is still under controversy but many researchers claim that Shilajatu exuding from the rocks of mountains is basically derived from vegetative source.^[9] Shilajathu mainly classified in to Gomutra Gandhi Shilajathu and Karpooa Gandhi shilajathu as per most of the Rasa Classics. Sooryathapi Method is adopted for purification of Gomutra Shilajathu using Triphala Kwatha. Purified Shilajathu was subjected to Classical as well as modern analytical methods.

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