

Research Article

A PHARMACEUTICO-ANALYTICAL STUDY OF DIFFERENT SAMPLES OF *SHILAJIT***Kavita Kishore^{1*}, Soniya Kumari², Anuradha²**

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KEYWORDS:*Shilajit*, *Shodhana*, Analysis.**ABSTRACT**

Shilajit (*Asphaltum punjabianum*) is an important and widely used drug in Ayurveda. *Shodhana* of *Shilajit* was widely mentioned but the methods of analysis were restricted. The composition of *Shilajit* mainly depends on the variety of plants that are associated with the rocks. Even though the precise origin of *Shilajit* is unknown, its popularity is increasing in today's society. Despite being commonly used, there is a lack of data regarding the analytical profile of *Shilajit*. **Aim:** An effort has been made to purify both natural and market samples as per AFI and analyze both raw and *Shudha Shilajit* samples, study their composition, and create the data to help generate the standard parameters of the *Shudha Shilajit* which will help to reduce the research gap of *Shilajit* analysis.

INTRODUCTION

Recently, there has been a noticeable increase in the use of various herbomineral medicines, which has raised concerns about their quality. For these medicines to be effective, they must be authentic, safe, potent, and non-toxic. Although classical methods of drug preparation typically ensure high-quality products but lack of standardization has led to a decline in the quality of available medicines, compromising their safety and efficacy. Classical texts of *Rasa Shastra*, have mentioned different methods for analyzing the quality of *Shudha Shilajit*. These methods though reliable and time-tested, are inadequate for assessing chemical composition for global standards. Disparities in *Shilajit* arise due to differences in its origin and market availability. Recent advancements in instrumental analysis techniques allow for a precise understanding of a mineral's elemental composition and also help to detect and prevent adulterations and substitutions in the pharmaceutical process.

In this study, the collection of natural samples of *Shilajit* from known sources in the Himalayan range

of Shimla and Kullu district and their purification processes were done. After that, both raw and *Shudha* samples of *Shilajit* were analyzed and compared on organoleptic characters, classical physicochemical parameters and modern parameters and data was prepared.



Fig. 1: Natural *Shilajit* in raw form collected from the site

MATERIALS AND METHODS**Materials****Collection of raw materials**

1. Raw natural *Shilajit* I was procured from Nirmand, Distt. Kullu and raw Natural *Shilajit* II were procured from Taklech, Dist. Shimla.
2. All three raw market *Shilajit* samples were procured from three different local vendors.

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Methods

1. Preparation of *Triphala Kwatha*
2. *Shodhana* of *Shilajit*

Preparation of *Triphala Kwatha*

Apparatus used- Weighing machine, *Khalva Yantra*, S.S container, S.S ladle, muslin cloth, measuring glass jar.

Procedure

Kwatha was prepared according to *Sharangdhara Samhita Madhyam Khand*^[2] by mixing the coarse powder of the *Dravya* with water in a ratio of 1:16. The mixture was boiled at *Madhyamagni* till it reduced 1/8th of its original volume. Then decoction was filtered through a single folded muslin cloth and collected as *Triphala Kwatha*.

*Note- For every *Shilajit* sample *Triphala Kwatha* was freshly prepared at the time of *Shilajit Shodhana*.

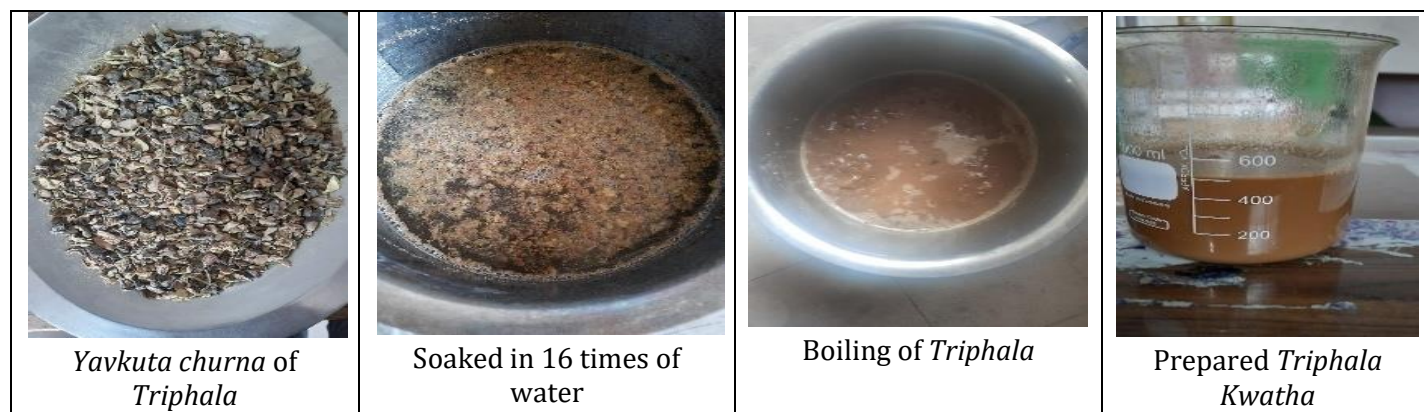


Fig. 2: Preparation of *Triphala Kwatha*

RESULT

Sample Name	Wt. of Yavkut <i>Triphala</i> (gm)	The initial quantity of water (ml)	<i>Triphala Kwatha</i> Obtained (ml)	Color of <i>Triphala Kwatha</i>	Taste of <i>Triphala Kwatha</i>
RSJN I - Raw <i>Shilajit</i> Natural Sample I	300	4800	570	Brownish	Astringent, sour
RSJN II- Raw <i>Shilajit</i> Natural Sample II	300	4800	590	Brownish	Astringent, sour
RSJM I- Raw <i>Shilajit</i> Market Sample I	300	4800	620	Brownish	Astringent, sour
RSJM II- Raw <i>Shilajit</i> Market Sample II	300	4800	580	Brownish	Astringent, sour
RSJM III- Raw <i>Shilajit</i> Market Sample III	300	4800	610	Brownish	Astringent, sour

Shodhana* of *Shilajit

Ashudha Shilajit- 1000gm

Triphala Kwatha- 500ml

Hot water- 2lt

Equipment used- Weighing machine, *Khalva yantra*, LPG stove, S.S vessels, S.S ladle, muslin cloth, S.S trays, digital thermometer, glass containers.

Procedure

Shilajit Shodhana was done according to A.F.I Part-1^[1]. *Ashudha Shilajit* was accurately weighed and ground into coarsely powdered form and added to a vessel containing a solution of *Triphala Kwatha* and hot water. This solution was heated on a *Mandagni* and then allowed to settle for three to four hours. After that solution was stirred vigorously till *Shilajit* dissolved completely. It was kept for sedimentation overnight. The next day supernatant liquid was decanted and stored, whereas residual material was collected separately. In residual material, 2L of hot water was added, mixed well, and kept undisturbed overnight. The next day the supernatant

liquid was kept separately. The decantation and maceration process was repeated till the decanted matter became clear (free from sand, and stone particles). The solution was heated on mild heat at 40°-50°C till a thick layer formed over the mixture and attained a semi-solid consistency then poured into an S.S. tray and placed in a drying shed till it became a viscous mass.



Fig. 3: Showing Method of Preparation of Shilajit Shodhan

RESULT

Sample Name	Quantity of Ashudha Shilajit (in gm)	Yield of Shudha Shilajit (in gm)	% Loss	Color of Shudha Shilajit	Odor and Appearance of Shudha Shilajit	Taste of Shudha Shilajit	Total time taken
SJN I	1000gm	149gm	85.1%	Blackish brown	Gomutra Gandhi soft, semi-solid	Kshaya, Lavana, Madhura	47 days
SJN II	1000gm	131gm	86.9%	Blackish brown	Gomutra Gandhi soft, semi-solid	Kshaya, Amla, Madhura	41 days
SJM I	1000gm	466gm	53.4%	Blackish brown	Gomutra Gandhi soft, semi-solid	Tikta, Kshaya, Madhura	52 days
SJM II	1000gm	266gm	73.4%	Blackish brown	Gomutra Gandhi soft, semi-solid	Kshaya, Tikta, Lavana, Madhura	43 days
SJM III	1000gm	136gm	86.4%	Blackish brown	Gomutra Gandhi soft, semi-solid	Tikta, Kshaya, Amla	41 days

***Note-** SJN I – Shilajit sample I, SJN II- Shilajit sample II, SJM I- Shilajit sample I, SJM II- Shilajit sample II, SJM III - Shilajit sample III



Fig. 4: End product of all the samples

Analysis of Shilajit

In the present study, Organoleptic and physicochemical analysis of *Shilajit* was carried out at the DTL, Joginder nagar.

Table 1: List of analytical parameters done for Raw and Shudha Shilajit samples

Organoleptic characters	Physiochemical parameters	Sophisticated instrumental parameters
Appearance	pH value	WD- XRF (Wavelength Dispersive - X-ray Fluorescence)
Color	Loss on drying	
Odor	Total solid	
Taste	Total ash	
	Acid insoluble ash	
	Water soluble extractive	

Table 2: Comparative Organoleptic and Physicochemical Parameters of Raw Shilajit Samples

Parameter	RSJN I	RSJN II	RSJM I	RSJM II	RSJM III	Mean	Range
Appearance	Solid piece	Solid piece	Solid piece	Solid piece	Solid piece	-	-
Color	Greyish	Greyish	Black	Greyish black	Greyish black	-	-
Odor	Go-Mutra	Go-Mutra	Go-Mutra	Go-Mutra	Go-Mutra	-	-
Taste	Bitter	Bitter	Bitter	Bitter	Bitter	-	-
pH	7.47	8.02	7.50	7.06	7.69	7.54	7.06-8.02
LOD	0.04%	0.08%	0.06%	0.04%	0.06%	0.05%	0.04%-0.08%
Total Solid	99.96%	99.92%	99.94%	99.96%	99.94%	99.94%	99.92%-99.96%
Total Ash	91.39%	96.70%	65.19%	93.44%	64.78%	82.22%	64.78%-93.44%
Acid insoluble ash	70.06%	88.40%	20.95%	82.28%	42.86%	60.91%	20.95%-88.40%
Water soluble extractive	13.92%	32.61%	54.48%	11.27%	1.96%	22.84%	1.96%-54.48%

Note- RSJN I - Raw *Shilajit* Natural sample I, RSJN II - Raw *Shilajit* Natural sample II, RSJM I - Raw *Shilajit* Market sample I, RSJM II Raw *Shilajit* Market sample II, RSJM III - Raw *Shilajit* Market sample III

Table 3: Comparative Organoleptic and Physicochemical Parameters of *Shudha Shilajit* Samples

Parameter	SSJN I	SSJN II	SSJM I	SSJM II	SSJM III	Mean	Range
Appearance	Viscous mass	Viscous mass	Viscous mass	Viscous mass	Viscous mass	-	-
Color	Dark brown	Dark brown	Dark brown	Dark brown	Dark brown	-	-
Odor	<i>Go-Mutra</i>	<i>Go-Mutra</i>	<i>Go-Mutra</i>	<i>Go-Mutra</i>	<i>Go-Mutra</i>	-	-
Taste	Bitter	Bitter	Bitter	Bitter	Bitter	-	-
<i>Jala Pariksha</i>	Comply	Comply	Comply	Comply	Comply	-	-
<i>Agni Pariksha</i>	Comply	Comply	Comply	Comply	Comply	-	-
pH	5.64	5.39	5.13	5.49	5.05	5.34	5.05- 5.64
LOD	35.63%	24.77%	24.14%	23.30%	22.66%	26.1%	22.66%-35.63%
Total Solid	64.37%	75.23%	75.86%	76.70%	77.34%	73.9%	64.37%-77.34%
Total Ash	22.92%	24.09%	25.34%	23.61%	15.82%	22.35%	15.82%-25.34%
Acid insoluble ash	1.56%	2.31%	1.24%	2.29%	1.42%	1.76%	1.24%-2.29%
Water soluble extractive	59.25%	66.80%	68.96%	63.68%	67.92%	65.92%	59.25% - 68.96%
Yield%	14.9%	13.1%	46.6%	26.6%	13.6%	22.96%	-

Note- SSJN I - *Shudha Shilajit* Natural sample I, SSJN II *Shudha Shilajit* Natural sample II, SSJM I - *Shudha Shilajit* Market sample I, SSJM II - *Shudha Shilajit* Market sample II, SSJM III - *Shudha Shilajit* Market sample III

Table 4: Elemental composition; XRF; Raw Samples

Analyte (%)	RSJN I	RSJN II	RSJM I	RSJM II	RSJM III	Mean	Range
O	72.89	74.59	33.66	68.20	71.96	64.26	33.66 74.59
Si	10.65	8.99	0.90	14.11	13.94	9.71	14.11 - 0.90
Al	5.10	3.99	0.10	4.56	4.32	3.61	0.10 -5.10
Fe	2.96	2.16	1.64	2.81	1.38	2.19	2.96 -1.38
K	2.11	2.29	5.82	1.00	0.97	2.43	0.97 -5.82
S	1.75	2.12	3.72	0.57	0.23	1.67	3.72 - 0.23
Na	1.74	1.24	0.18	3.29	3.49	1.98	0.18 -3.49
Mg	1.69	1.60	0.17	1.72	0.90	1.21	0.17 - 1.72
Cl	0.43	0.87	11.40	0.90	0.41	2.80	0.41- 11.40
Ca	0.30	1.73	40.73	2.10	1.98	9.36	0.30- 40.73
Ti	0.18	0.14	0.33	0.17	0.10	0.18	0.10 -0.33
P	0.06	0.18	0.80	0.33	0.11	0.29	0.06 - 0.80
Cr	0.01	0.02	-	-	0.03	0.02	0.01- 0.03
Zr	0.01	0.01	-	0.02	0.01	0.01	0.01- 0.02
Zn	0.01	-	-	0.01		0.01	0.01
Rb	0.01	0.01	-	0.00	0.00	0.01	0.01
Co	0.00	-	-	-	-	-	-
Mn	0.00	0.03	-	0.04	0.02	0.02	
Ni	0.00	-	-	-	-	-	-
Sr	0.00	0.01	0.36	0.03	0.03	0.10	0.01 - 0.03
Y	0.00	0.00		0.00	-	-	-
Nb	0.00	-	-	-	-	-	-
Cu	-	0.01	-	0.01	0.00	0.01	0.01

Ba	-	-	-	0.03	-	-	0.03
Pb	-	-	-	0.00	-	-	-
Br	-	-	0.13	-	-	-	0.13

Table 5: Elemental Composition; XRF; Shudha Shilajit Samples

Analyte (%)	SSJN I	SSJN II	SSJM I	SSJM II	SSJM III	Mean	Range
O	39.42	45.09	36.36	45.09	56.46	44.48	56.46-36.36
Ca	25.06	20.77	31.98	20.77	19.21	23.55	19.21- 31.98
K	17.06	10.68	8.87	10.68	6.37	10.73	6.37 - 17.06
S	11.66	7.05	12.28	7.05	5.19	8.64	5.19- 12.28
Cl	1.99	10.05	8.43	10.05	4.29	6.96	1.99- 10.05
Fe	1.46	2.68	0.23	2.68	2.99	2.00	0.23- 2.99
P	1.55	1.78	1.25	1.78	2.68	1.80	1.25- 2.68
Si	0.91	1.07	0.08	1.07	0.96	0.81	0.08 - 1.07
Na	0.26	0.21	0.19	0.21	0.49	0.27	0.19 - 0.49
Al	0.23	-	-	-	0.25	0.24	0.23 - 0.25
Mg	0.19	-	0.08	-	0.22	0.16	0.08 - 0.22
Sr	0.14	0.24	0.10	0.24%	0.29	0.25	0.10 - 0.29
Br	-	-	0.09	-	-	-	0.09
Mn	-	-	-	-	0.31	-	0.31
Cu	-	-	-	-	0.21	-	0.21

Table 6: Analytical Parameters with a range of Shudha Shilajit findings may be considered as helping tools for further work

Parameter	Standardization of Shilajit	Range of Observation in the study
Appearance	Viscous mass	Viscous mass
Color	Dark brown	Dark brown
Odor	Gomutra	Gomutra
Taste	Bitter	Bitter
Jala Pariksha	Comply	Comply
Agni Pariksha	Comply	Comply
pH (10% aq Solution)	5.05- 5.64%	5.05 - 5.64%
LOD	NMT 35.63%	22.66-35.63%
Total solid	NLT 64.37%	64.37-77.34%
Total ash	NMT 25.34%	15.82-25.34%
Acid insoluble ash	NMT 2.29%	1.24-2.29%
Water soluble extractive	NLT 59.25%	59.25 - 68.96%

Elemental Composition of Shudha Shilajit

The elemental composition may confirm the presence of around 10 elements out of the following elements; (the range of percentage is not confirmatory; may be considered for further studies and a helpful tool for *Shilajit* analysis).

Table 7: Elemental composition of Shudha Shilajit with a range of findings (XRF)

Element in Shudha Shilajit	Range of observations in the study (%)
O	36.36-56.46
Ca	19.21- 31.98
K	6.37 - 17.06
S	5.19- 12.28
Cl	1.99- 10.05
Fe	0.23- 2.99

P	1.25 - 2.68
Si	0.08 - 1.07
Na	0.19 - 0.49
Al	0.23 - 0.25
Mg	0.08 - 0.22
Sr	0.10 - 0.29
Br	0.00 - 0.09
Mn	0.00 - 0.31
Cu	0.00 - 0.21

DISCUSSION

Shilajit is one of the *Maharasa* explained in *Rasa Ratan Samucchaya*^[3]. *Shilajit Shodhana* was carried out as per the reference of A.F.I Part-1 and the *Agnitapi* method was adopted. For this, each sample of raw *Shilajit* was taken in 1kg and powered in coarse form, and mixed with *Triphala Kwatha* and hot water. After that solution was kept for sedimentation till the supernatant liquid became clear. Then solution was heated till a thick creamy layer appeared on a semi-solid paste. After that transferred in S.S. trays and placed in a drying shed. The process took 47 days, 41 days, 52 days, 43 days, and 41 days respectively to obtain *Shudha Shilajit*. The total yield of *Shudha shilajit* was 14.9%, 13.1%, 46.6%, 26.6%, and 13.6% respectively for each sample.

The five samples of *Shudha Shilajit* have shown organoleptic characteristics like blackish brown in color, *Gomutra* smell, Soft in touch, semisolid in appearance, and *Kshaya*, *Madhura*, *Lavana*, *Amla*, and *Tikta* in taste. All the *Shudha* samples were subjected to classical parameters (*Jala Pariksha* and *Agni Pariksha*) and showed positive tests.

The range of physico-chemical analysis of *Shudha Shilajit*- pH was 5.05-5.64, LOD was 22.66-35.63%, total solid was 64.37-77.34%, total ash was 15.82-25.34%, acid insoluble ash was 1.24-2.29%, water soluble extractive was 59.25-68.96%.

Major elements found in *Shudha Shilajit*- the mean weight of oxygen 44.48%, calcium 23.55%,

potassium 10.73%, sulphur 8.64%, chlorine 6.96%, iron 2.00%, and phosphorus 1.80% respectively.

Minor elements found – the mean weight of silica 0.81%, Na 0.27%, strontium 0.25%, aluminium 0.24%, and magnesium 0.16% respectively.

CONCLUSION

We can conclude that on going through classical texts, the *Agnitapi* method was used because of its easy handling and feasibility. The yield of naturally procured *Shilajit* was approximately 13-15% and for market samples is approx. 30%. *Shudha Shilajit* was acidic. On analysis, no finding of toxic elements was found.

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