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PHARMACEUTICO –ANALYTICAL STUDY ON *ASHUDHA* AND *SHUDHA SHILAJATU*

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ABSTRACT

Background: *Shilajatu* is considered one of the wonderful medicines of Ayurveda. Neither a plant nor animal substance, it is a mineral pitch that oozes from the rocks of Himalayas, as they become warm in the summer month. *Shilajatu* is a blackish brown exudation of variable consistancy found in the serene surroundings of Himalayas. *Shilajatu* has been given prime importance by the *Acharyas*.

Materials and methods: *Shodhana* is a process by which impurities or poisonous materials of the substance are removed. The impure *Shilajatu* contains stony particles, sand, mud, wooden fibres and several metallic impurities leading to various diseases. So purification of *Shilajatu* is very essential. After purification it becomes brittle.

Results: Total yield of *SS* was 40%. In the elemental analysis it showed the major presence of iron denoting the *lohadrija shilajatu*. Other analytical parameters like FTIR, XRD, HPTLC were done to both the samples.

Conclusion: The sample taken for *Shodhana* was genuine in both ancient and modern parameters.

Keywords: *Ashudha Shilajatu, SS, Sodhana*, FTIR, XRD, HPTLC.

Abbrevations: *AS- Ashudha shilajatu, SS-Shudha Shilajatu*

INTRODUCTION: *Shilajatu (Asphaltum punjabinum)* is an exudation from rock 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). Ayurveda mentions it as wonderful medicine. It describes that *shodhita Shilajatu* after *bhavana*(addition of constituents of a drug to it for a disease to be cured in form of watery extract etc.) can cure even the *asadhya* diseases and disorders. Nearly all the ancient and medieval texts, which constitute the frame of Ayurveda strongly, upheld the curative action and therapeutic properties of the drug. The *Shilajatu* has been in extensive use in the preparations of a number of medicines and their utility has been most dependable because of this very constituents. In bruhatraee acharya says "there is hardly any curable disease which cannot be controlled or cured with the aid of *Shilajatu*."[1]*Shilajatu* is the exudation caused by the intense heat of the sun (in the month of *jyeshta and ashadha*) from the Himalayan rocks in the womb of which lie deposited gold and other metals[2]. Similar explanations are seen in other *rasa granthas* also.

OBJECTIVES:

- To do *Shodhana* of *ashudha shilajatu*
- To do physico chemical analysis of *ashodhita* and *shodhita shilajatu*.

MATERIALS AND METHODS: Before administration of any drug, it is well instructed that the drug should undergo through a proper purification procedure so that it should be devoid of not only the external impurities and internal impurities, but also it will have good potentiating effect too. Therefore it is essential that the drug should be well purified to avoid any untoward effect in the human beings.

TEST: The tests mentioned in Ayurvedic texts are only of a crude type. Genuinety of any drug is justified by the testing it as per the specification. In *Samhitas and Rasarnava* there is no specific description about the test of *Shilajatu*. The testing techniques which are following:

The drug is supposed to be authentic if -

i. *Shilajatu* is put on fire it erects perpendicularly and burn without smoke.

ii. If pure *Shilajatu* is put in water through the tip of a thin erect glass it will come down slowly after spreading like fibre.

iii. The pure *Shilajatu* should contain the smell of cow urine[13].

In the present study *Shodhana* of *shilajatu* is carried out by *suryatapi* method[21]

Ingredients: 1.*Ashudha shilajatu*=2 Kg.2. *Triphala Kwatha* = 1litre

3. Hot water = 4litre

Procedure:

- *A.S* was weighed and made into coarse powder in a clean *Khalwa yantra*.
- Take a stainless steel clean and wide mouth vessel.
- Powdered *AS* was put into the vessel, to this 1ltr of warm *Triphala Kashaya* was added and stirred well.
- At the end 4ltr of hot water was added and stirred well so that *shilajatu* gets dissolved in the liquid.
- The mixture was kept undisturbed in hot sunrays for 3hrs.
- After 3hrs the undisturbed mixture was macerated well and filtered. The external impurities like sand, mud, etc were filtered and the supernatant was kept in another vessel.
- To the solid filtrate obtained again some quantity of hot water was added and kept in sunlight.
- The collected supernatant liquid was kept in strong sunlight for *suryatapi* method of *Shodhana*.
- The container was covered with a lid every day after sunset.
- The creamy layer so formed was collected, which is *SS*.
- Hot water was added periodically with a gap of 3-5 days.
- The process was carried out for 2months.

OBSERVATION:

- It took around 15-20 mins for the complete mixing of the ingredients.
- The color of the mixture was dark brownish black.
- After 3hrs the solid filtrate was complete undissolved mud particles.
- The color of supernatant liquid was Dark brown in color.
- The collected *SS* was sticky and pitch black in color.
- The collected *shilajatu* was dried in shade to remove the left over moisture.

Precautions:

- The *suryatapi* method of *Shodhana* should be carried in strong sunrays, preferably in summer.
- The container should be clean and wide mouthed.
- It is better to place the container over the terrace where there is sufficient sunlight and process is carried out without any obstacles.
- The lid of the container should be closed every day after sunset, so that it is not affected by dust, dried leaves, etc.; falling into the container.
- Sufficient quantity of hot water should be added so that there is adequate space for layer formation.
- The so formed creamy layer of *Shilajatu* should be collected carefully without discutbing the supernatant fluid.

RESULTS: *Shilajatu Shodhana*

Initial weight of *shilajatu*: 2000 g

Wt of *shilajatu* after *Shodhana*: 800g

Loss : 1200g:Total yield : 40%

Table .1: Showing classical Parameters for Analysis of SS

TEST	**OBSERVATION**
Varna	*Krishna varna*
Rasa	*Kashaya, Tikta.*
Sparsha	*Shlakshna*
Gandha	*Gomutra gandha*
When put on fire	*Lingakara* without any smoke
Put in water	Settled down in the form of strings

Table .2: Showing organoleptic characters of A.S and S.S.

Physical test	**A.S**	**S.S**
Colour	Reddish –brown	Blackish – brown
Odour	Characteristic	Characteristic
Taste	Astringent ,salty, bitter	Astringent, salty
Appearance	Rough powder	Sticky lump.

Table .3: Showing result of Physical Tests of A.S and S.S

Parameters	**A.S**	**S.S**
pH	7.08 $\pm$ 0.10	5.38 $\pm$ 0.10
Total Ash value	33.25%	29.00%
Acid Insoluble Ash	17.50%	15.10%
Water Soluble Ash	63.00%	69.50%
Loss on Drying	3.50%	10.50%

Table .4: Showing result of Chemical tests of A.S., S.S

Contents	**A.S**	**S.S**
Total Iron	1.83%	1.58%
Ferrous	1.67%	1.39%
Ferric	0.16%	0.19%
Copper (in ppm)	952	323
Gold (in ppm)	2571	2135
Silver (in ppm)	250	215

X-Ray Diffraction Results:

Table .5: Showing the chemical composition, crystal structure of A.S, S.S

Parameters	A.S.	S.S
Name	Aluminium oxide, Magnesium oxide, Ferric oxide, Sodium chloride, Silicon	Ferric oxide, Calcium oxide,Magnesium oxide Nitrogen, Silocon
Composition	Al_20_3, MgO, Fe_2O_3, NaCl, Si	Fe_2O_3,CaO,MgO,N_2,Si
Crystal system	Cubic,cubic,cubic,cubic	Cubic, Cubic, Hexagonal,cubic

Table .6: Showing FT-IR Observations of A.S

Transmittance Peak Frequency Cm^{-1}	Standard Peaks Frequency Cm^{-1}	Specific Type Of Bond	Bond	Functional Group
3309.40	3330–3270	Narrow, strong	–C≡ C–H: C–H stretch	alkynes (terminal)
2931.31	3000–2850	Medium	C–H stretch	Alkanes
1619.65	1710–1665	Strong	C=O stretch	α,β–unsaturated aldehydes, ketones
1523.45	1550–1475	Strong	N–O asymmetric stretch	nitro compounds
1450.66	1470–1450	Medium	C–H bend	Alkanes
1373.43	1480-1350	Variable	-C-H bending	Alkanes
1286.22	1300–1150	Medium	C–H wag (–CH 2 X)	alkyl halides
1242.16 1149.51	1250–1020	Medium	C–N stretch	aliphatic amines
1026.22	1400-1000	strong	C-F stretch	Alkyl Halide
817.99 767.43 715.67	850–550	Medium	C–Cl stretch	alkyl halides
574.13	690–515	Medium	C–Br stretch	alkyl halides

Table. 7: Showing FT-IR observations of S.S

Transmittance Peak Frequency Cm^{-1}	Standard Peaks Frequency Cm^{-1}	Specific Type Of Bond	Bond	Functional Group
3365.64	3600-3200	strong, broad	O-H(stretch, H-bonded)	Alcohol
2925.41	3000–2850	Medium	C–H stretch	Alkanes
1620.50	1650–1580	Medium	N–H bend	1° amines
1524.13	1550–1475	Strong	N–O asymmetric stretch	nitro compounds
1453.10	1500–1400	Medium	C–C stretch (in–	Aromatics

			ring)	
1376.64	1470–1450	Medium	C–H bend	Alkanes
1286.52	1335–1250	Strong	C–N stretch	aromatic amines
1242.22 1151.00 1076.71 1024.10	1250–1020	Medium	C–N stretch	aliphatic amines
823.04 765.24 712.82	850–550	Medium	C–Cl stretch	alkyl halides
603.24 526.50	690–515	Medium	C–Br stretch	alkyl halides

HPTLC

Table.8: R_f values of all the sample AS

At 254 nm	At 366 nm	After Derivatisation
0.03(L Green)	0.03(F L Violet)	-
-	0.05(F L Violet)	0.05(L Violet)
-	0.08(F L Violet)	-
0.10(L Green)	-	-
-	0.12(F L Violet)	0.12(L Violet)
0.15(L Green)	0.15(F L Violet)	-
-	-	0.18(L Violet)
0.20(L Green)	0.20(F L Violet)	-
-	0.23(F L Violet)	-
-	0.28(F L Violet)	-
0.32(D Green)	0.32(F L Violet)	0.32(L Violet)
-	0.41(F L Violet)	-
0.45(L Green)	-	-
-	0.48(F L Violet)	-
0.53(L Green)	-	-
-	0.56(F L Violet)	-
-	0.59(F L Violet)	-
0.64(L Green)	0.64(F L Violet)	-

***L-Light, F-Fluoroscence**

Table .9: R_f values of all the sample SS

At 254 nm	At 366 nm	After Derivatisation
0.03(L Green)	0.03(F L Violet)	0.03(Violet)
0.06(L Green)	-	0.06(Violet)
0.09(L Green)	0.09(F L Violet)	0.09(L Violet)
-	-	0.11(Violet)
-	0.14(F L Violet)	-
-	-	0.16(L Violet)

0.18(L Green)	0.18(F L Violet)	
-	-	0.21(L Violet)
-	0.26(F L Violet)	0.26(L Violet)
-	0.29(F L Violet)	-
0.33(Green)	0.33(F L Violet)	0.33(L Violet)
-	0.38(F L Violet)	-
0.42(L Green)	-	0.42(L Violet)
-	-	-
0.46(L Green)	0.46(F L Violet)	-
0.52(L Green)	0.52(F L Violet)	-
	0.58(F L Violet)	-
0.64(L Green)	0.64(F L Violet)	-
0.71(L Green)	-	-
-	0.73(F L Violet)	0.73(L Violet)
0.77(Green)	0.77(F Violet)	-
0.82(L Green)	0.82(F Violet)	0.82(L Violet)

***L-Light, D-Dark, F-Fluoroscence**

PRIMARY PHYTOCHEMICALS:

Table .10: Results of preliminary phytochemical tests for A.S and S.S

Test	**A.S**	**S.S**
Carbohydrate	Absent	Absent
Protein	Absent	Absent
Alkoloid	Absent	Absent
Flavanoids	Present	Present
Tannin	Present	Present
Terpenoid	Present	Present
Steroid	Present	Present

DISCUSSION: In classics *shilajatu* has been greatly acclaimed not only as *rasayana* but also the curer of the hardly curable disorders even. It has been included in different groups by different authors. Though the classics have enumerated various varities of *shilajatu* but commercially only two types are available. Various researchers have worked on analytical and biolgical effects of *shilajatu* and reported that it contains Humic acid, Fulvic acid etc.

Shodhana media used was *triphala*. The probable reason for this may be because of its properties of Tridosha shamana (especially Kapha) that is why it is added in *Shilajatu* and it augments the effect in reducing lakshanas of Medoroga.

Shodhana was done by doing *Shoshana* in *suryatapa*. Here the supernatant creamy layer of *shuddha shilajatu* gets collected. *Shilajatu Shodhana* with *triphala kwatha* was done. 2kg *AS* was mixed with 1 litre of hot *triphala kwatha* and 4litre of hot water. It was not sufficient for the process. It required 6-8 times of hot water to extract pure *shilajatu* from its impure form. The formation of creamy layer was very slow and thin layer was formed this might be because of the unavailability of good sample or less heat of sun for the

extraction. It was observed that only 40% *Shodhita shilajatu* was obtained. Better yield of *SS* can be obtained if the season is good and good quality of sample is procured.

Discussion on analytical parameters: The *lingakara lakshana* was seen and this maybe because of the presence of fulvic acid in the sample. *SS* was analyzed for the classical parameters.

The P^H of **A.S and S.S** are **7.08 & 5.38 respectively.** All the values indicate the weak acidic nature of the drugs. The dissociation of molecules is highly temperature dependent. This might be the reason for the increase in the pH of A.S compared to S.S. Total Ash value of **A.S, S.S** and are **33.25%** and **29.00%** respectively. As per the pharmacopeia standards end product SS contains least ash value and is stable more bioavailable than AS. The acid insoluble ash of **A.S., S.S** were **17.50%** and 15.10**%** respectively. As **S.S** possess very least amount of acid insoluble ash & on par with standards, which signifies that a considerable amount of drug is soluble in the acidic media of stomach. It also rules out the contamination of drug with silicates. The water soluble ash of **A.S., S.S** were 63.00% and 63.00% respectively. This signifies that water is the soluble media. **A.S, S.S** Possessed 3.50**%** and 10.50% loss on drying at 110^0C. Hence it can be stated that **A.S** has least amount of moisture content, and **S.S** is having moisture content which maybe because of the process followed. The drug is having hydroscopic activity so it should be stored in air tight container. **Total** percentage of iron in **A.S** is 1.83**%,** 1.67**%** of which is in ferrous form, 0.16**%** is in ferric form. **S.S** contains 1.58**%** of total iron in which 1.39% is in ferrous form and 0.19% in ferric. In the forms of iron ferrous form is bioassimable. As seen in **S.S** 1.39% of ferrous is present which makes the drug more potent.

The X-ray Diffraction studies of **A.S** and **S.S** was done to know the structure and chemical composition of the samples. According to XRD reports the chemical compositions of samples is discussed below. Totally 18 peaks were identified in **A.S** at different angels (2 θ) from 14.242 to 94.326. Peaks of **A.S** confirmed the presence of Aluminium oxide , Magnesium oxide, Ferric oxide, Sodium chloride and Silicon

SS showed 4 peaks following peaks were chosen as strong with their relative Intensity and compared to standard. . Peaks of the sample confirmed the presence of Magnesium Oxide, having Cubic crystal structure; Nitrogen, having hexagonal crystal structure;

FTIR analysis of A.S., S.S-reveals the presence of many functional groups which affirms the entity of herbal compounds used for the *Shodhana*. **A.S**. comprises of functional groups like Alkynes, Alkanes, Nitro, Alkyl halides and aliphatic amines. The functional groups identified in **SS** are alcohol,aromatic amines, Aliphatic amines and alkyl halides. The presence of aliphatics, cyclic and acyclic compounds denoted the presence of humic acid,benzoic acid and fulvic acid in the taken samples.

HPTLC is the sophisticated analytical parameter for the evaluation of the herbal drugs. HPTLC can also serves as Fingerprinting technique for identification and quantification of the herbal and herbo-mineral formulations. Through HPTLC technique major phytochemical present the drug or formulation can be estimated. HPTLC of AS and SS showed same peaks.

CONCLUSION: The best method for the *shodhana* of *shilajatu* is by *suryatapi*

method as the volatile oils or contents present in it do not get destroyed .From the above discussion it can be concluded that the so taken *shilajatu* showed good analytical reports denoting the presence of inorganic and organic components stating the taken sample is genuine.

PHOTOS:

Raw shilajatu

shilajatu coarse powder

Preparation of Triphala kashaya

Ingredients for shodhana

Mixing

shilajatu kept undisturbed

Maceration after 3 hours

Day -1

Day- 10

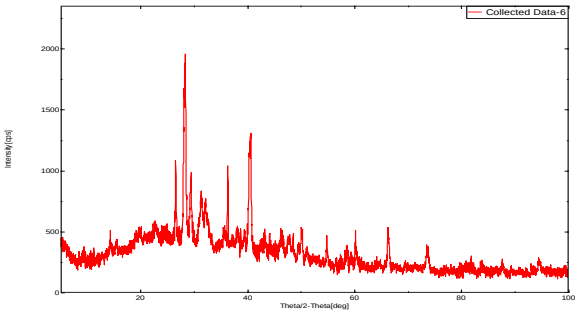
Day-40

Shodhita Shilajatu

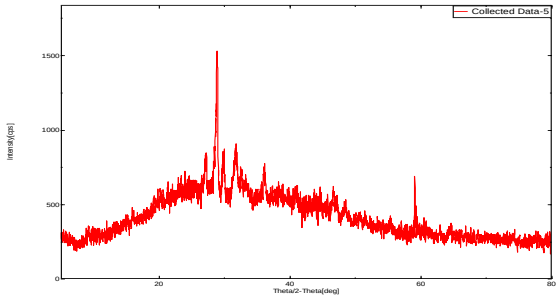
Lingakara lakshana of Shilajatu

GRAPHS:

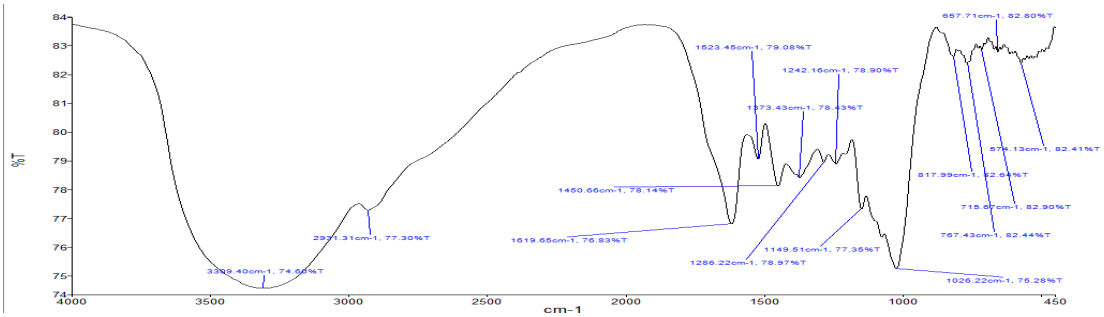
XRD Spectra of AS



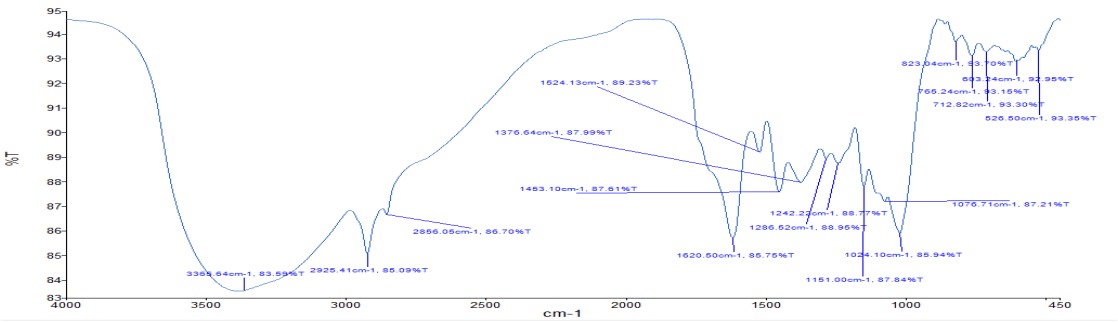
XRD Spectra of SS

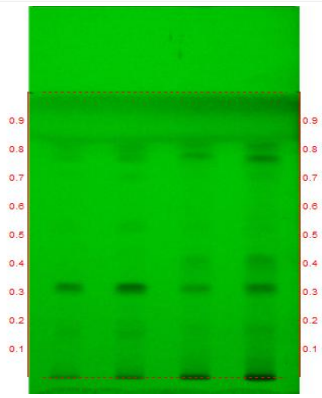


F TIR –ASHODHITA SHILAJATU



FTIR –SHODHITA SHILAJATU





HPTLC image

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