



Research Article

PHARMACEUTICO-ANALYTICAL STUDY OF *SHODHITA SHILAJATU*

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ABSTRACT

In the Pharmaceutical process of different forms of medicines raw material & its quality plays an important role. During ancient time *Vaidyas* were themselves used to collect the raw materials herbs based on their occurrence and organoleptic characters, i.e., typical taste, texture, smell, color, and there types after selection they utilized them in preparing medicines. Based on their own observations, principles of drug processing and ideal quality of finished product, etc., have been documented by them. The principles were developed based upon the scientific parameters prevailing in those days. Materials and Method: Raw *Shilajatu* was subjected to *Shodhana* and *Shodhita shilajatu* was subjected to analytical study. The required materials for the process were collected from the Pharmacy of BLDEA's AVS Ayurveda Mahavidyalaya pharmacy Vijayapur, analytical lab. Raw drugs were purchased from Dorle and Sons retailer from Kolhapur Maharashtra. *Shilajatu shodhana* was carried out according to reference mentioned in *Rasaratna samuchaya*. Analytical tests concern Indian Pharmacopeia methods were followed. Result: Raw *Shilajatu* took 5 ½ hour to 6hour in *Shodhan* procedure in both batches. Three days for drying. In analytical tests physical constants like pH ash value, solubility, specific gravity, moisture content etc result values were under normal limit.

KEYWORDS: *Shodhita shilajatu*, Pharmaceutical study, Analytical study.

INTRODUCTION

Shilajatu is considered as *Rasa dravya* categorized under *Maharasa* by *Rasa vagbhata*.^[1] It is indicated for many diseases including *Sthoulya*, *Kusta*, *Prameha*, *Medogna*.^[2] *Shilajatu* is having multidimensional therapeutic activities mentioned in different classics like immuno modulation, Anti microbial, hypo lipidemic, spermatogenic, positive results in latrozol induced PCOS (Poly Cystic Ovarian Syndrome) and hypoglycemic.^[3]

In spite if this Acharaya Charak has coated that

Na

soasthirogobhuvishadhyarupashilahvayanajayetprashyl tatkalyogevidhibhiprayuktaswasthsyachorchavipulad adati".^[4]

The administrations of *Shilajatu* at proper time with proper *Anupana* cure almost all the curable diseases in the world. Though *Maran*, *Satwapatana* procedures are being mentioned to *Shilajatu*^[5] but in almost all preparations as an ingredient use of *Shodhit shilajatu* is recommended and it is followed.

Proper following up of pharmaceutical methods in *Shodhana* procedure only leads to its

production of good quality of *Shodhita shilajatu*. This will have good therapeutic values. Improperly following up of procedure can affect its therapeutic values. Hence to validate its pharmaceutical procedures and analyse its qualities, pharmaceutico-analytical study was conducted.

MATERIALS AND METHODS

Method of Preparation of *Shodhita Shilajatu*

Raw *Shilajatu* (4kg), *Triphalakwatha* (16 litres) and *Goghrita* to smear on plate during drying the *Shodhit shilajatu* were used for the purification of *Shilajatu*. *Shilajatuchurna* was prepared in *Khalwayantra* Powdered *Shilajatu* was poured & dissolved in *Triphalakwatha* with constant stirring. This Mixture was subjected to heating by keeping it on stove. *Mandagni* (low flame) was maintained during the procedure along with constant stirring the mixture. When mixture started to attain thick consistency it was poured on ghee smeared plate & kept for drying in shadow. After conforming that *Shilajatu* is completely dried and then it was removed from plate, powdered and stored in container.

Analytical Study^[6,7]

a. Solubility test: A pinch of *Shodhita shilajatu* powder was taken in a dry test tube with one ml of solvent and shaken for one minute. Then observed for solubility, non-solubility and sparingly solubility. Solubility test was conducted with the following solvents. Distilled water, Xylene, Benzene, Toluene, Acetone, Hydrochloride acid, Ethylealchol, Methanol, Chloroform, Carbon Tetrachloride

b. Percentage of Solubility: 100ml of distilled water + 5gm of *Shodhita shilajatu* powder was stirred well, kept for 24 hours and filtered through Whatman's filter paper no. 42. It was kept for drying and weighed the filter paper with residue after complete drying. Then percentage of solubility was calculated as per standard formula.

c. Determination of Moisture Content: A clean and dry petridish was weighed. 5gm of dried *Shodhita shilajatu* powder was taken in the petridish and weighed. Petridish was kept in hot air oven for an hour at 105°C. After one hour the petridish containing *Shodhita shilajatu* was taken out of hot air oven and weighed. Again the petridish was kept in hot air oven for one more hour at 105°C. The procedure was repeated till the constant weight of petridish containing powder was obtained.

Difference in weight × 100 Percentage of Moisture content = Weight of sample

d. Determination of Specific Gravity: Empty picnometer was weighed (w1) and picnometer was filled with distilled water weighed (w2). Afterwards took 1% solution prepared by taking one gram of *Shodhita shilajatu* dissolved in 100 ml of distilled water & weight measured (w3). All these three weights noted.

$W3 - W1 / W2 - W1$

e. Determination of pH Value: Digital handy pH meter was calibrated by using standard buffers solution of known pH 4.0 and 9.2 at 30°C. The reference electrode was thoroughly washed with distilled water every time and water was drained by using filter paper. 1% solution of *Shodhita shilajatu* by taking 1gm of *Shodhita shilajatu* in 100ml of distilled water was prepared then the tip of electrode was completely immersed in the solution of *Shodhita shilajatu* and pH value was recorded.

f. Ash value: A clean and dry Silica crucible was weighed. 5gm of sample of *Shodhita shilajatu* was weighed accurately and transferred to the Silica crucible. The Silica crucible along with sample was weighed. It was subjected to incineration in a

electric muffle furnace till carbon free ash was obtained maintaining 450°C temperature. At the end Silica crucible was taken out from the muffle furnace allowed to cool and weighed.

Percentage of Ash = Difference in weight × 100 / Weight of sample

g. Determination of Water Insoluble Ash: Total ash obtained by the above procedure boiled with 25ml of distilled water for five minutes and cooled. It was filtered through Whatman filter paper no. 40 and insoluble matter was collected. Dried and ignited the filter paper along with insoluble matter cooled and weighed. The percentage of water insoluble ash was calculated as follows: weight of filter paper with ash - weight of filter paper / Wt of ash × 100

h. Determination of Acid Insoluble Ash: Total ash obtained by ash value procedure boiled with 25 ml of dilute HCl. Insoluble matter was collected on ashless filter paper (Whatman's Filter Paper 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 / Wt of ash × 100

Phytochemical Constituents of *Shodhita shilajatu*: *Shodhita shilajatu* was subjected for extraction with distilled water. Then the concentrated extract was used for phytochemical screening to observe the phytochemical constituents.

a. Method of Extraction: About 50gms of *Shodhita shilajatu* powder was subjected for extraction with distilled water from soxhlet extractor. The extracts was concentrated by distilling the distilled water at low temperature and then dried on a water bath. Aqueous extract was subjected for phytochemical analysis.

b. Detection of Alkaloids: Sol. of Extract + Wagner's reagent (K.I.Soln.) Reddish brown ppt indicates the presence of Alkaloids. Reddish brown coloured ppt was observed in the aqueous extract of *Shodhita shilajatu* That indicates the presence of alkaloids

c. Detection of Carbohydrates: Benedict's test (Test for reducing sugars): 5ml Benedict's reagent was heated in a test tube, 8-10 drops of test sol. Was added. Then brick red ppt indicates presence of carbohydrates. Brick red ppt was observed in the aqueous extract of *Shodhita shilajatu* that indicates the presence of carbohydrates.

d. Determination of Tannins: Sol of Extract + FeCl₃ (Ferric chloride) Blue colour ppt was not observed in the aqueous extract of *Shodhita shilajatu* that indicates the absence of tannins.

- e. Determination of Steroids:** Libermann Burchard sterol reaction: Sol of Extract + conc. H₂SO₄ + Chloroform. Blood red ppt was not observed in the aqueous extract of *Shodhita shilajatu* that indicates the absence of steroids.
- f. Determination of Triterpenoids:** Salkowski's test: Sol of Extract + conc. H₂SO₄ + Chloroform (10ml chloroform + 90ml distilled water i.e. 1:9) Yellow ppt was observed in the aqueous extract that indicates the presence of Triterpenoids.
- g. Determination of Flavanoids:** Sodium hydroxide solution test: Sol of Extract + 10% lead acetate sol. Yellow ppt was not observed in the aqueous extract of *Shodhita shilajatu* that indicates the absence of Flavanoids.
- h. Detection of proteins:** Millions Biuret test: Sol of Extract + 2ml 10% NaOH sol. + 2ml 10% CuSO₄. Blue colour ppt was not observed in the aqueous extract of *Shodhita shilajatu* that indicates the absence of Proteins.
- i. Detection of Saponins:** Foam index test: Sol of Extract + 20ml D/W shaken for 15 minutes. Foam was observed in the aqueous extract of *Shodhita shilajatu* that indicates the presence of saponins.
- j. Detection of Carotenoids:** Sol of Extract + 85% H₂SO₄. Blue colour at the junction of two layers was not observed that indicates the absence of Carotenoids.

RESULTS

Pharmaceutical Study

Shodhana of *Shilajatu* was carried out in two batches. In first batch 2 kg of *Shilajatu* and 8 liters of *Triphalakwatha* used. In the first batch procedure it took 6hour. Later on allowed to dry under shade. After complete drying *Shodhita shilajatu* was dried weighed & stored. It was noted that out of 2 kg 1.8kg was obtained and 0.2kg loss was noted. Similar procedure followed in the 2nd batch too and at the end 1.8 kg *Shodhita shilajatu* obtained. In the second batch pharmaceutical procedure took 5 hour 30 min.

Analytical Study

Shodhita shilajatu tested for organoleptic characteristics. Appearance concerned it was amorphous powder form, black in color, taste concern *Kashayarasa* and odor concerned it was similar to *Gomutra*.

Table 1: Showing Solubility of *Shodhita shilajatu*

S.No	Solvent	Solubility
1	Distilled Water	S
2	Methanol	NS
3	Ethyl alcohol	NS
4	Petroleum ether	NS
5	Acetone	NS
6	Benzene	NS
7	Toluene	NS
8	Chloroform	NS
9	Xylene	NS
10	Carbon tetrachloride	NS

NS = Not soluble, S = soluble

Table 2: Showing Physico-chemical standards of *Shodhita shilajatu*

S.No	Physical constants	Results
1	pH value	4.9
2	Specific gravity	0.999
3	Moisture content	1.3
4	Ash value	17.2 %
5	Water insoluble ash	1.1 %
6	Acid insoluble ash	14.1%
7	% of solubility	76%

Table 3: Showing phyto-chemical constituents of Shodhita shilajatu

S.No	Solvent	Solubility
1	Alkaloids	-Ve
2	Carbohydrates	-Ve
3	Tannins	+Ve
4	Steroids	+Ve
5	Triterpenoids	+Ve
6	Flavanoids	+Ve
7	Proteins	+Ve
8	Saponins	+Ve
9	Carotenoids	+Ve

Table 4: Shows Elements in their Oxides present in Shodhita Shilajatu^[8]

Major Oxides	Percentage of Oxides
MnO	16.2%
CuO	15.7%
Rb ₂ O	14.7%
ZnO	12.5%
Moderate Oxides	Percentage of Oxides
PbO	5.14%
PbO	5.14%
MnO	5.14%
Average Oxides	Percentage of Oxides
SiO ₂	2.88%
P ₂ O ₅	2.33%
MgO	2.18%
Na ₂ O	2.08%
CaO	1.08%
SO ₃	0.776%
K ₂ O	0.468%

Table 5: Shows Elements present in Shodhita Shilajatu^[8]

Major elements	Percentage of Oxides
Rb	14.7%
Zn	12.5%
O	8.07%
Moderate elements	Percentage of Oxides
Al	5.14%
Pb	5.14%
Average elements	Percentage of Oxides
Si	2.88%
P	2.33%
Mg	2.18%
Na	2.08%
Fe	1.25%
Ca	1.08%
S	0.776%
Cl	0.725%
K	0.468%

DISCUSSION

Pharmaceutical study was carried out in two batches. Each time 2 kg of *Shilajtu* and 8 liters of *Triphalalkwatha* was used in *Shodhana* procedure. In the first batch it took almost 6 hours to complete the procedure and to pour on plate. Whereas second time it took 5 hour & 30 minutes. During the boiling process of the mixture of *Triphalalkwatha* and *Shilajatu*, initially boiling up of *Kwatha* was observed. Gradually decrease in the level of *Kwatha* was seen due to evaporation of *Kwatha* after boiling. The consistency of *Kwatha* too gradually changed to thick. When consistency becomes thick it was poured on vessel and when it got dried completely then collected from plate and powdered. Its weight was noted and it was found that 10% loss recorded in both batches. Powdered *Shilajatu* was stored in airtight container.

Analytical Study

Shodhita shilajatu was tested for organoleptic characters. Appearance concerned it was amorphous powder, color concerned dark color, odor concerned gomutra odor, taste concerned *Kashaya* & *Gomutra* taste, touch concerned it was soft.

Shodhita shilajatu was tested for physical constants test. pH was 4.9, Specific Gravity was 0.999, moisture content was 1.3%, ash value 17.2 w/w, water insoluble ash 1.1%, acid insoluble ash 14.1% and 76% of solubility was noted.

Subjected to solubility test in various chemical solvents like Distilled water, methanol, ethyl alcohol, petroleum ether, acetone, benzene, toluene, chloroform, xylene, carbon tetrachloride. It was found soluble in distilled water only & in other remaining chemical solvents it was not soluble.

On subjecting to phytochemical constituents analysis study it has shown positive for, Tannins, Steroids, Triterpenoids, Flavanoids, Proteins, Saponins, Carotenoids and negative for Alkaloids, Carbohydrates.

In the elemental analysis following oxides were found in major, Moderate & Average percentages those are major oxides MnO, CuO, Rb₂O, ZnO Moderate Oxides PbO, MnO Average Oxides SiO₂, P₂O₅, MgO, Na₂O, CaO, SO₃, K₂O.

In the same way following metals were found in major, Moderate & Average percentages those are major elements Rb Zn O Moderate elements Al Pb Average elements Si P Mg Na Fe Ca, S, Cl, K In the research work carried out by Dr Shashirekha.^[8]

CONCLUSION

In the present study it was found 10% loss was observed in pharmaceutical study. In analytical study physical constants were under normal limit. Tannins, Steroids, Triterpenoids, Flavanoids, Proteins, Saponins, Carotenoids were detected in phytochemical constituents analysis.

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Raw Triphala



Raw Shilajatu



Triphalakhwatha



Shilajatu dissolved in Triphalakhwatha



Filtering Shilajatu dissolved in Triphalakhwatha



Shodhita Shilajatu kept for drying



Shodhita Shilajatu