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 samhaya. 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 vaghbata.[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 quoted that "Na soasthirogobhvisadhyarupashilahvayanajajyetrprashyaltatkalyogvidhibhirayuktaswasthsyachorchavipulad 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 Shirajatu

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, Hydrochloric 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.

\[
\text{Difference in weight} \times 100 \% \ \text{Percentage of Moisture content} = \text{Weight of sample}
\]

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.

\[
W_3 – W_1 / W_2 – W_1
\]

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 300°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 an 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.

\[
\text{Percentage of Ash} = \frac{\text{Difference in weight} \times 100}{\text{Weight of sample}}
\]

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 X 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:

\[
\text{Weight of filter paper with ash} - \text{weight of filter paper} / \text{Wt of ash} \times 100
\]

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 were 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 shacked 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 6 hour. 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

| Major Oxides | Percentage of Oxides |
|--------------|----------------------|
| MnO          | 16.2%                |
| CuO          | 15.7%                |
| Rb2O         | 14.7%                |
| ZnO          | 12.5%                |
| Moderate Oxides | Percentage of Oxides |
| PbO          | 5.14%                |
| PbO          | 5.14%                |
| MnO          | 5.14%                |
| Average Oxides | Percentage of Oxides |
| SiO2         | 2.88%                |
| P2O5         | 2.33%                |
| MgO          | 2.18%                |
| Na2O         | 2.08%                |
| CaO          | 1.08%                |
| SO3          | 0.776%               |
| K2O          | 0.468%               |

### Table 5: Shows Elements present in Shodhita Shilajatu

| 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 Shilajatu and 8 liters of Triphalakwatha 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 Triphalakwatha 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 powered. 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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Triphala

Shilajatu

Triphalakwatha

Shilajatu dissolved in Triphalakwatha

Filtering Shilajatu dissolved in Triphalakwatha

Shodhita Shilajatu kept for drying

Shodhita Shilajatu