RESEARCH PAPER

Enhancement of Fulvic Acid in Shilajitu via Shodhana with Triphala Kwatha: A Quantitative Titrimetric Study

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ABSTRACT
Ayurveda emphasizes on prevention as well as cure of the disease simultaneously. Prevention of a disease can be achieved by using drugs which are in general terms called as Rasayana. These include herbal as well as mineral drugs. Out of the mineral group of Rasayana, Shilajitu a well-known drug has occupied its position as a unique drug. It is commonly used as a rejuvenator, and is also useful in many urinary disorders. As it is a Rasa Dravya, which is non-toxic in nature, classics have mentioned its Shodhana (purification) by various methods to remove its possible impurities if any. One such method of Shodhana is mentioned by the use of Triphala Kwatha which was carried out in the current study. Which was aimed to evaluate the role of Triphala Kwatha in Shodhana (purification) of Shilajitu by assessing the amount of fulvic acid before and after Shodhana (purification). Ashuddha Shilajitu was purified in Triphala Kwatha by dissolving it, followed by evaporation of mixture. The obtained Shuddha (purified) Shilajitu sample was then evaluated by Titrimetric method. The amount of fulvic acid after Shodhana was increased almost eleven times as compared to Ashuddha sample. Hence, we can say that Shodhana of Shilajitu not only helps to remove the impurities but also enhances its efficacy by increasing the amount of Fulvic Acid.

Keywords: Ayurveda, Shilajitu, Fulvic Acid, Shodhana, Triphala Kwatha, etc.
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INTRODUCTION

According to classical literature of *Ayurveda*, *Shilajitu* is an exudate mineral that drips out of mountainous rocks in the hot season[1] on the other hand *Rasashastra* classics considers it as one of the *Maharasa*[2]. *Charaka* in his treatise has included *Shilajitu* in a group of *Rasayanas.* Wherein he has mentioned several types of *Shilajitu* viz. *Swarna Shilajitu*, *Rajata Shilajitu*, etc[3]. *Ayurvedic* literature is thus filled with many references regarding *Shilajitu* and is considered to be a source of strength, effective on a broad variety of diseases[4,31], including urinary conditions, diabetes, bone fragility and overall weakness[4,30]. The Sanskrit etymology, *Shilajitu*, literally means conqueror of the mountains and destroyer of the weakness[4]. This demonstrates the significance with which *Shilajitu* was imported and practiced to preserve the state of activity and life-span. According to different *Rasashastra* texts, *Shilajitu* is divided in two types according to the smell viz. *Gomutra Gandhi* (smelling like cow urine) and *Karpura Gandhi* (smelling like camphor)[5]. The present study was done for *Gomutra Gandhi Shilajitu*.

Contemporary scientific research of *Shilajitu* or Black Bitumen describes it to be a complex organo-mineral compound that includes humic materials, fulvic acid, dibenzo-alpha-pyrone, minerals, and trace elements[6]. So far, it is thought among those the fulvic acid is the primary bioactive component that permeates most of the pharmacological performances of *Shilajitu*[7]. Fulvic Acid is reported to have antioxidant[8], anti-inflammatory, immunomodulatory[9], and neuroprotective activity[10].

Shilajitu is, however, never utilized as it is in natural form. Usually, soil, residual plants, microbial wastes and heavy metals are found in it naturally[11]. Classical *Ayurvedic* pharmaceutics also insists on the *Shodhana* (purification) process prior to its use in medicine. *Shodhana* does not only aims to eliminate toxic or undesirable substance, it is also meant to augment bioavailability and the strength of drug[12]. There are various procedures of *Shodhana* stated in

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classical literature, and one of them is purification in *Triphala Kwatha*[13], which is probably a common practice.

Triphala, is a classical *Ayurvedic* concoction, commonly taken as *Rasayana*[14]. A decoction of this also called as *Triphala Kwatha* which offers an acidic media, rich in phenols, that makes impurities easy to dissolve and may chemically alter the *Kwatha* components as well[15].

In previous studies, *Shodhana* has been shown to have desirable qualities of enhancing the safety status of *Shilajitu* through lessening heavy metals and the microbial burden[16]. There have also been studies where post-purification, solubility has been found to increase and a greater level of therapeutic activity been observed[17]. However, the specific anthropometric change in the concentration of fulvic acid after *Shodhana* in a specific media, especially in the form of quantitative analysis is limited. As fulvic acid is an indicator of pharmacological effect, the measurement of this parameter in *Shilajitu* is significant in confirming that the classical claim is legitimate.

The current study was taken to determine the influence of *Shodhana* of *Shilajitu* in *Triphala Kwatha* to see the possible changes in amount of fulvic acid in *Shilajitu*. The analysis was conducted by a conventional titration procedure to estimate the content of fulvic acid. Comparative analysis was also done between *Ashodhita Shilajitu* (unpurified) and *Shodhita Shilajitu* (purified using *Triphala Kwatha*).

MATERIALS AND METHODS

Source of *Shilajitu*

Raw *Shilajitu* is in exudate form, which was gathered from authentic sources. The substance was a black resin that was slightly brown in colour and sticky soft in nature. The crude was not treated to any extent. For the purification process *Triphala Kwatha* was used.

Step-by-Step *Shodhana* Procedure (Table: 1)

Table:1 Shodhana of Shilajitu

Step	Process	Activity and Observations
1	**Media Preparation**	*Triphala Kwatha* was prepared by boiling 1 part of coarsely powdered *Triphala* in 16 parts of water until it was reduced to 1/8th, then filtered[18].
2	**Dissolution**	100g of raw *Shilajatu* was added to the warm *Triphala Kwatha* and stirred continuously to ensure complete solubility.
3	**Filtration**	The mixture was filtered through a clean muslin cloth to remove insoluble impurities like sand, stones, and plant debris.
4	**Settling**	The filtrate was allowed to cool and settle, effectively isolating the bioactive resin from the media.
5	**Drying/Evaporation**	The purified material was kept in a controlled environment until it reached a constant weight of 90g.
6	**Final Collection**	The resulting *Shodhita Shilajatu* was smooth, homogenous, and deep black with a shiny texture.

Figure1 Ashuddha Shijajitu[19]

Figure 2 Shuddha Shilajitu[19]

Determination of Fulvic Acid Content

Principle

The estimation of fulvic acid was done using a conventional titrimetric method[20]. This was done by oxidation of fulvic acid using potassium permanganate ($KMnO_4$) under the influence of indigo sulfonic acid indicator. This is directly proportional to the amount of $KMnO_4$ that is used in consumption and the quantity of fulvic acid in the sample.

Reagents

Distilled water, hydrochloric acid (0.1 N), indigo sulfonic acid solution (indicator), potassium permanganate 0.02M, Whatman filter paper.

Procedure

All procedures were conducted in the Vasu Research Centre (A Division of Vasu Healthcare Pvt. Ltd., Vadodara, Gujarat, India), an AYUSH-approved Ayurvedic Testing Laboratory (License No. GATL/08). Universal laboratory conditions were provided to achieve accuracy and reproducibility of the procedures.

Preparation of samples:

A correct weight of 0.1 gm of sample (*Ashodhita* and *Shodhita Shilajitu* were taken individually) was taken in a 1000 ml conical flask. 50 ml of distilled water was turned over till the sample was completely dissolved. Then, 1 ml of 0.1 N HCl was put and the solution was sonicated for 15 minutes. Whatman filter paper was used to filter the solution with the residue being washed with 25 ml distilled water. A mixture of the obtained filtrate was mixed with distilled water to total the volume to 750 ml. As an indicator 10 ml of solution of indigo sulfonic acid was added. The solution was incubated by adding 0.02 M $KMnO_4$ solution until blue to golden yellow coloration occurred. Before every sample estimation, a blank titration was done.

Calculation:

Fulvic acid was calculated in percentage based on the following formula:

$$\% \text{ of Fulvic acid} = \frac{(BR\text{-}B) \times F \times M}{\text{----------}} \times 100$$

0.02 x W

Where:
BR = burette reading (sample)

B = blank burette reading

F = factor (0.001701)

M = actual molarity of KMnO4

W = mass of the substance taken (g)

Replication

The analysis of both *Ashodhita* and *Shodhita* samples was done in triplicate and average values were recorded in order to be able to get accuracy.

RESULTS

It was observed that fulvic acid content in *Shilajitu* before and after purification using *Triphala Kwatha* showed a strong variation between the two samples of the product.

Physical Observations

This table summarizes the weight loss attributed to the removal of physical impurities and the *Shodhana* process. (Table: 2)

Table 2: Yield and Quantitative Change

Parameter	Weight	Percentage	Observation
Initial Weight (Ashodhita *Shilajitu*)	100 g	100%	Contains sand, rocks, and fibrous material.
Final Weight (Shodhita *Shilajitu*)	90 g	90%	Pure, bioactive resin enriched with Fulvic Acid.
Weight Loss	10 g	10%	Attributed to the removal of non-medicinal physical impurities.

These properties describe the sensory characteristics of the substance before and after the purification process (Table 3).

Table 3: Organoleptic Properties of Shilajatu

Property	Before *Shodhana* (Raw)	After *Shodhana* (Purified)
Appearance	Irregular, stony, or resinous masses	Smooth, lustrous, thick paste or dry powder
Color	Dark brown to blackish-brown	Deep black with a glossy sheen
Odor	Strong, distinct smell (resembling cow urine)	Characteristic odor, but refined and less pungent
Taste	Bitter, astringent, and slightly acrid	Bitter and astringent (*Tikta* and *Kashaya Rasa*)
Touch	Sticky, rough, and contains grit	Soft, smooth, and homogeneous

These values represent the chemical stability and purity levels achieved through the *Shodhana* process (Table 4).

Table 4: Physicochemical Parameters

Parameter	Typical Value (Purified)	Significance
pH (1% aqueous solution)	5.0 – 6.5	Indicates slight acidity/stability
Water Soluble Extract	> 80%	High solubility indicates better bioavailability
Total Ash	10% – 20%	Reflects the mineral content
Acid Insoluble Ash	< 2%	Low values indicate removal of sand/silica
Moisture Content	8% – 12%	Ensures shelf-life and prevents microbial growth
Specific Gravity	1.1 – 1.8	Confirms the density of the purified resin

Chemical Analysis

Fulvic acid percentage was calculated using standard titrimetric measurement as mentioned before. The results are as follows (Table 5).

Table 5. Fulvic acid content of Shilajitu before and after Shodhana in Triphala Kwatha

Sample type	Fulvic acid (%)
Ashodhita Shilajitu	0.76 %
Shodhita Shilajitu	8.34 %

It was revealed that the percentage of fulvic acid content was 0.76 in the unpurified sample and 8.34 after purification. This is an estimate of about ten- to eleven-fold increase in the content of fulvic acid.

Replication and Accuracy

The estimates were made in triplicate and the difference between codes of replicates was low (+ - 0.05%). This consistency made the method reliable and the utilization of fulvic acid rise reproducible.

Key findings

Triphala Kwatha (as a *Shodhana* media) led to a significant increment in the amount of fulvic acid in *Shilajitu*. It was also done to enhance the organoleptic properties (appearance, odour, and texture) of the material. The rise was reproducible and was even observed in replicate tests.

DISCUSSION

In the current experiment, it was shown that purification of *Shilajitu* in *Triphala Kwatha* causes significant change in the Fulvic acid content which is 0.76% in the unpurified

sample and 8.34% in the purified sample. This result corresponds to the *Ayurvedic* classical concept of *Shodhana* effectively not only in excluding impurities, but also to improve the quality of raw drug.

Role of *Triphala Kwath* in the Transformation

Triphala Kwatha is a media that is used for purification of many metals and minerals. It is enriched with high concentrations of tannins, gallic acid, ellagic acid, ascorbic acid and many other polyphenolic compounds[21], which lead to its antioxidant, antimicrobial and chelating effect[22]. *Triphala* also acts as a solubilising agent that aids in the dissolution of the impurities and isolating the inert components with the bioactive ones[23]. Due to the presence of polyphenols in *Triphala* it is able to chelate heavy metals eliminating toxic load. It can also be predicted that *Triphala* can help bring complex organic matter present in *Shilajitu* to hydrolyse into smaller and more bioavailable fragments like fulvic acid[24]. It is also possible that due to certain phyto-chemicals of *Triphala* form a complex compound with *Shilajitu* and stabilize the fulvic acid or even raise its concentration. Such increment in the level of fulvic acid may both be explained as the sum total due to elimination of contaminants and the addition of fulvic acid by the method of hydrolysis and oxidation throughout the process of purification.

Significance of Fulvic Acid

Fulvic acid is a water soluble low molecular weight component of humic acid that is formed naturally through the natural decay of vegetable matter[25]. It is complex in structure and consists of polyphenolic and carboxylic groups which have endowed with multiple biological functions[10].

Possible reasons of increased amount of Fulvic Acid

The increase in the amount of fulvic acid obtained after *Shodhana* of *Shilajitu* in *Triphala Kwatha* may be because of one of the following reasons. *Triphala* consists of tannins, gallic acid, ellagic acid, ascorbic acid and phenolic compounds which act as strong natural chelating and extracting agents. During boiling in *Triphala Kwatha*, these compounds may break down the high molecular weight humic substances into low molecular weight fulvic acid[7]. *Triphala Kwatha* is slightly acidic in nature (pH around 3)[22] which may have caused acidic hydrolysis by breaking down protein carbohydrate mineral complexes[26]. This breaking down then leads to degradation of larger polymer chains ultimately increasing free Fulvic Acid concentration in *Shuddha Shilajitu*. *Triphala* contains Ascorbic acid which acts as a reducing agent[15]. Hence is able to convert higher molecular humates in simpler fulvic acid molecules[27]. The adaptogenic and energy-enhancing potential of *Shilajitu* further supports the clinical relevance of ensuring a high fulvic acid content post-purification.[28,29]

CONCLUSION

This study shows definite evidence that the conventional *Shodhana* procedure is much more than a mere cleaning procedure of the raw *Shilajitu*. *Triphala Kwatha* which is used as a medium of purification, shows defined results of an elevation in the fulvic acid levels. Precisely, the concentration increases to 8.34% (nearly eleven times) in the final purified resin as compared to 0.76% in the crude sample. This probably can be attributed to the acidic nature of *Triphala* and polyphenolic compounds which may be involved in breaking down high molecular weight humic substances into smaller fragments by hydrolysis and oxidation. This quantitative study finally confirms the classical *Ayurvedic* beliefs that ideal pharmaceutical procedures are necessary to optimize the therapeutic potential, safety and bioavailability of *Shilajitu*.

Conflict of interest - None

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