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PHYSICO-CHEMICAL STUDY OF SHILAJIT WITH ARJUNA KWATH BHVITA & KHADIR KWATH BHAVITA

Sanjay Kumar Kanoujiya[1*], Satya Prakash Chaudhary[2] and Neeraj Kumar[3]

[1]MD Scholar of Deptt. of Rasa Shastra Faculty of Ayurved, IMS,BHU Varanasi-221005.
[2]Ph. D Scholar of Deptt. of Dravyaguna, Faculty of Ayurved IMS, BHU Varanasi-221005.
[3]Professor of Deptt. of Rasa Shastra Faculty of Ayurved, IMS,BHU Varanasi-221005.

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***Corresponding Author**
Sanjay Kumar Kanoujiya
MD Scholar of Deptt. of Rasa Shastra, Faculty of Ayurved IMS, BHU Varanasi-221005.

ABSTRACT

Arjuna and khadir both are very important medicinal plant in ayurveda according to samhita and nighatu and according to rassashtra shilajit also very important minral durg in ayurveda. The composition of these drugs was used as antidiabetic action. For Shilajeet, an important Ayurvedic drug, shodhana and bhavana has been prescribed and different methods are available for it, shodhana by using Gomutra and Triphala kwatha,hot water etc and bhavana with other specific material use to treatment of various disease, being two of them.for the preparation of formulation uwe used the arjuna and khadir kwath as a bhavana drugs. In present study Of Shilajit with Arjuna Kwath Bhvita & Khadir Kwath Bhavita have been taken for physical–chemical analysis in terms of loss on drying, ash values, Acid insoluble ash values, Water soluble ash value etc and Assay of elements X- Ray flouoresence, Determination of pH, TLC Chromatogram of different sample of Bhavita Shilajatu etc.

KEYWORDS: *Terminalia arjuna*, *Acacia catechu*, Shilajit, Ash value, TCL.

INTRODUCTION

In ancient days the drugs were prepared by the physician himself with the help of experienced assistants in the small pharmacy attached to his clinic over the centuries, the communication gap has tremendously reduced resulting in the large scale manufacturing and wide distribution of Ayurvedic drugs at national and international level. The increasing need for drugs have made it necessary that some sort of uniformity in the manufacturing of Ayurvedic medicines should be brought about. The need has also been felt for statutory

control to ensure standards of Ayurvedic drugs in the modern sense. Considering the vast number of such drugs and heir formulations, is a time and money consuming tasks and will take considerable time for its achievements.

Chemical study ensures not only chemical constituents but also tells us standards of any preparation. It not only gives the standards of the product but indirectly gives suggestions for further advancement if required.

In the corporative era there is an exponential growth in the field of herbal and Ayurvedic medicine in the last few decades. It is getting popularized in developing as well in developed countries owing to its natural origin and side effects. In olden times, Vaidyas used to treat patients on individual basis, and prepare medicines according to the need of the patient. But the situation has changed now, Ayurvedic and herbal medicines are being manufactured on the large scale in Pharmaceutical units, where manufacturers come across many problems such as availability of good quality raw material, authentication of raw material, availability of standards, good standardization methodology of single drugs and formulation, quality control parameters.[1] *Terminalia arjuna* commonly known as arjuna is an belong to the family *combrutaece* and acacia catachu commonly known as khadir is belong to the family Leguminosae. It is used by the local practitioners for the treatment of various disorders. The present study was conducted for the evaluation of inorganic element and heavy metal using XRF.

MATERIALS AND METHODS[2,3,4,5,6,7,8]

To evaluate the quality of finished products it becomes necessary to subject the drugs for chemical study put the drugs in the prospect of science. The drugs which are manufactured should be well understood and interpreted in the light of modern chemistry to provide proper scientific background. Shodhana is an important process mentioned in Ayurveda which should be carried out before using in number of products. Various methods has been prescribed for shodhana of different materials. For Shilajit, an important Ayurvedic drug, shodhana and bhavana has been prescribed and different methods are available for it, shodhana by using Gomutra and Triphala kwatha, hot water e.t.c and bhavana with other specific material use to treatment of various disease, being two of them.The present study is the comparative evaluation of Arjuna kwath bhavita suddha Shilajit and Khadira kwatha bhavita suddha Shilajit samples. As mentioned earlier the capsules were prepared and

analysis of both the samples were carried out in Department of Rasa shastra & Bhaisjya kalpana and Department of Medicinal chemistry Faculty of Ayurved IMS.BHU Varanasi.

I. Qualitative analysis as per Ayurveda

II. Physico-chemical parameters

A Determination of pH

B. Loss on drying,

C. Ash value

D. Acid insoluble ash value

E. Water soluble ash value

F. Assay of elements

G. TLC Chromatogram of different sample of Bhavita Shilajatu.

1: Qualitative analysis as per Ayurveda: Two different Shilajit sample (Arjuna kwath bhvita & Khadir kwath bhavita) were taken for their qualitative analysis as per Ayurvedic text.

2: Physico-Chemical parameters

Determination of pH

10 gm sample was taken, to it 100 ml distilled water was added, extracted for 2 hours with occasional shaking and filtered. The pH of the filtrate was noted in a 'Systronics pH meter'.

Loss on drying: this test was performed to find out the moisture content of the sample.

1 g of exactly weighed sample was taken in a previously weighed petri dish and dried in oven at 110 c till constant weight. Then petri dish was taken out weighed after self cooling and forms the weight loss the percentage of loss on drying was calculated and expressed as %w/w.

Determination of Ash value

The ash value was determined by taking about 2 gm accurately weighed, sample in a crucible and subjecting it to incineration at 450° C in a muffle furnace, until it was freed from carbon. Later it was cooled and weighed. From the weight of residue the percentage of ash was calculated and expressed as % w/w.

Determination of acid insoluble ash

The ash obtained from above experiment was boiled with 25 ml of 6NHCl for five minutes, and the solution was filtered through ashless Whatman filter paper number 41. It was washed with hot water, ignited and weighed. From the weight of the ash obtained, the percentage of acid insoluble ash was calculated.

Determination of water soluble extractive

About 5 gm, accurately weighed, sample was extracted with 100 ml water by keeping it for about 20 hours and filtered. From the filtrate 20 ml is transferred to a previously weighed evaporating dish, evaporated to dryness on a water bath and dried completely by heating in an oven till constant weight. The percentage of water soluble extractive was calculated from the weight of the residue obtained and expressed in terms of % w/w.

Analytical data of Physico-Chemical analysis of sample

TLC: Identification of chemical can be detected by observation of spots of identical R_f value and about equal magnitude obtained, respectively, with an unknown and a reference sample chromatographed on the same plate. A visual comparison of the size and intensity of the spots usually serves for semi-quantitative estimation.[7,8]

Preparation of plates

Suspension of the coating substance was prepared in accordance with the instructions of the supplier and, using the spreading device designed for the purpose, a uniform layer of the suspension, 0.25 to 0.30mm thick, on a flat glass plate 20cm long was spread. The coated plates were allowed to dry in air, heated at 100ºC to 105ºC for at least 1 hour and allowed cooling, protected from moisture. The plates were protected from moisture and used within 3 days of preparation. At the time of use, the plates were again dried, if necessary, the distance of each spot from the point of its application was measured & recorded the R_f value were calculated by dividing the distance travelled by the spots by the distance travelled by the front of the mobile phase.

X- Ray fluorescence(XRF)

Introduction

X- Ray fluorescence is a powerful quantitative and qualitative analytical tool for elemental analysis of material it is ideally suited to the measurement of film thickness and composition, determination of elemental concentration by weight of solids and solutions, and identification

of specific and trace elements in complex sample matrices. Intensity x-rays fluorescent x-ray will be emitted from the sample at energy levels unique to those elements. The basic concept for all XRF spectrometer is a source of sample and detection system. The source irradiate the sample and the detector measure the fluorescence radiation emitted from the sample. In most cases for XRF, the source is an x-ray tube. Alternatives are radioactive source or a synchrotron.

Basic principle

Thus x-ray fluorescent principle is depicted in figure. An inner shell electron is excited by an incident photon in the x-ray region. During the de excitation process, an electron is moving from a higher energy level to fill the vacancy. The energy difference between the two shell appears as an x-ray emitted by the atom. The x-ray spectrum acquired during the above process reveals a number of character peaks. The energy of the peaks leads to the identification of the elements present in the sample (qualitative analysis), while the peak intensity provide the relevant or absolute elemental concentration (semi quantitative or quantitative analysis).

A typical xrf spectroscopy arrangement includes a source of primary radiation (usually a radio isdotope or an x-ray tube) and equipment for detecting the secondary x-rays.

XRF SOURCES

The irradiation of a sample is usually performed by radioisotopes sources or x-ray tubes (1-5). The energy of primary radiation should be higher than but close to the binding energy of the k and l shells electron of the excited atoms. the most wide spread radioisotope sorces include fe^{55}, Co^{57},Cd^{109}, am^{241}. These sources emit x-rays of definite energy, therefore beiong capable of efficiently exciting a limited number of atoms. As a result, to analyze a broad range of elements, a combination of radioisotope sources is necessary.

X-ray optics can be used to enhance both the types of XRF instrumentation. For conventional XRF instrumentation, typical focal spot sizes at the sample surface range in diameter from several hundred micrometers up to the several millimeters. Poly capillary focusing optics collect x-rays from the divergent x-ray source and direct them to a small focused beam the sample surface with diameters as small as ten of micrometers. The resulting increased intensity, delivered to the sample in a small focal spot, allows for enhanced spatial resolution for small feature analysis and enhanced performance for measurement of trace elements for

micro x-ray florescence application. Doubly curved crystal optics direct an intense micron sized monochromatic x- ray beam to the sample surface for enhanced elemental analysis.

X-ray detector

Solid state detectors have prevailed for the secondary x-rays measurement. In particular, Si(Li) and HPGe detectors, operating under liquid nitrogen temperature, are most commonly being used due to their high resolution.

CALIBRATION

Quantitative XRF analyze require calibration of the measuring arrangements, which may be performed by 2 major approaches

Empirical and fundamental parameters (FP) calibration.

The empirical calibration is based on the analysis of standards with known elemental compositions. To produce a reliable calibration model. The standard must be representative of the matrix and targeted elemental concentration ranges of the sample analyzed. Maintaining the same sample morphology (particle size distribution, heterogenicity and surface condition) and source sample geometry for both standard and sample measurement is essential in empirical calibration.

Alternatively "standard less" FP techniques may be used, which rely on builot in mathematical algorithm that describes the physics of the detector's response to pure element. In this case, the typical composition of the sample must be known, while the calibration model may be verified and optimized by one single standard sample.

DETECTION LIMITS

Two types of detection limits should be considered in XRF analysis

a. Instrument detection limits, which represent the threshold concentration of a given element that a particular instrument can resolve and
b. Method detection limits, related to the sample preparation and analysis time.
c. Depending on the element to be analyzed and the sample matrix, typically achieved detection limits vary between 10 and 100 ppm.

Sample preparation

Procedure for sample preparation vary considerably in the case of *In-situ* or intrusive measurements. Solid sample must be polished to assure surface homogeneity, while powders

are usually pressed into pellets. In all cases, x-ray transparent supporting media should be used (polyethylene, kapton, mayloar etc.

Aim and Object

To analyse element of three sample of Shilajatu of water purified Shilajatu (S1W) and Arjuna & Khadir akwath bhavita Shilajatu (S1WA &S2WK).and compare these with each other finding.

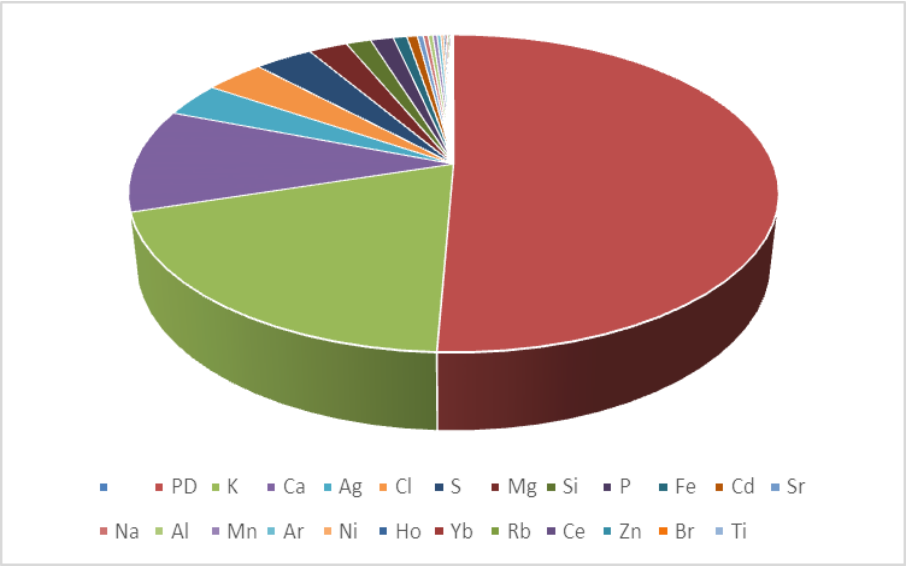
- Sample S1W and Sample S1WA
- Sample S1W and Sample S2WK

RESULT AND DISCUSSION

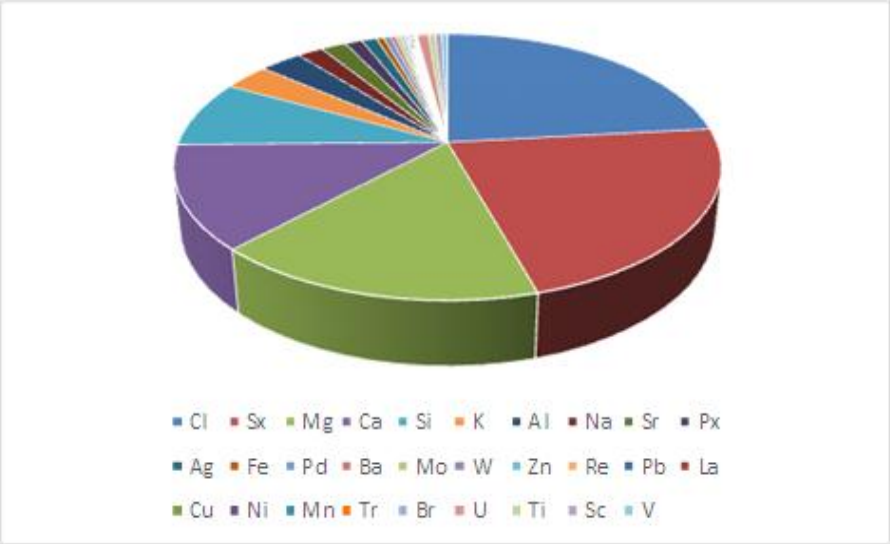
Varna(colour)	Krishna
Lustre	Gugguluabha
Odour	Gomutragandhi
Consistency	Mridu
Dissolving in Water	Dissolve with give a stream
Reaction on fire	Burn without fumes and give lingakarakriti

Parameters	**S1 (A.kw)**	**S2 (Kh.kw)**
pH.	5.10	5.26
Loss on drying	8.04%	9.03%
Ash value	18.76%	19.19%
Acid insoluble ash value	10.57%	11.70%
Water soluble ash value	84.66%	95.57%

Rf value	**A.kw S1**	**Kh.kw S2**
	0.33	0.51



Arjuna kwath



Khadir kwath

ELEMENT (S1WA)	WEIGHT (%)
PD	50.67
K	19.51
Ca	10.58
Ag	3.53
Cl	3.52
S	3.44
Mg	2.32
Si	1.47
P	1.36
Fe	0.812
Cd	0.62
Sr	0.352
Na	0.289
Al	0.278
Mn	0.254
Ar	0.219
Ni	0.154
Ho	0.13
Yb	0.116
Rb	0.11
Ce	0.1
Zn	0.08
Br	0.04
Ti	0.034

ELEMENT (S1WA)	WEIGHT (%)
Cl	26.91
Sx	23.24
Mg	18.68
Ca	13.04
K	4.32
Hg	2.63
Sr	2.35
Na	1.52
Si	1.46
Px	1.19
Ag	1.17
Fe	0.62
Pd	0.517
Mo	0.311
Au	0.300
Dy	0.23
Ar	0.21
Yb	0.200
Pt	0.179
Zn	0.163
Al	0.146
Pb	0.135
Mn	0.135
Ni	0.104
Tb	0.100
Br	0.83
Ge	0.52
Cr	0.38

From the above method we found that LOD value of the sample of S1WA and S2WK are 8.046, 9.043 respectively. These values show the moisture contained in the samples. S1WA have less LOD & S2WK area sample have maximum LOD, so S1WA sample contained less moisture and S2WK sample maximum moisture contained.

The total ash values of the sample of S1WA and S2WK are found 8.04, 9.03. The total ash value represents the inorganic salt, naturally occurring in drug and deliberately added to it as a form of adulteration. So according that value Dabur S1WA contained less inorganic salt and adulteration but S2WK sample maximum contained the water soluble ash value of S1WAsample is less than S2WK sample.

Acid insoluble ash values were found to be more for purified sample because it contains any siliceous material like sand, clay etc more than unpurified sample, due to addition of some siliceous materials. According to results the S1WA is less basic (pH-5.10) and the S2WK is highly basic (pH-5.26). The Rf value of sample indicate the purity of the sample. when the Rf value is higher then compound are pure .and when the Rf value is less then compound are impure. According to result the S1WA sample pure (0.33) and the S2WK impure (0.51).

Total of inorganic elements were determined in the both prepared samples were using X- Ray flouoresence (XRF). This shows the concentrations of various metals, in the analyzed prepared sample. The study revealed that all the metals do possess heavy metals but within the prescribed limit.

CONCLUSION

On the basis of the result the Arjuna & Khadira kwath bhavita Shilajatu (S1WA &S2WK) sample are good and the concentration of the heavy metals in the limit. Quality of both Sample is fair. All inorganic elements and he heavy metal were also in limit. therefore these sample are free from the heavy metal toxicity.

REFERENCE

1. Agarwal, A., Critical issues in Quality Control of Herbal Products, Pharma Times, 2005; 37(6): 0911.
2. “Indian Pharmacopoeia”, 2007; I.
3. Anonymous, “Indian Pharmacopoeia” Govt. Of India, Ministry of Health, Controller of Publication, Delhi, India., 1996.

4. C.K. Kokate, A.P. Purohit, S.B. Gokhate, Pharmacognosy, Shri D.K. Furia Nirali parkaspublication 14, Budhwar Peth, Jogeshwari Mandir Lane, Pune- 411 002 PP122; 1990.
5. Protocol of testing, Pharmacopeal Laboratory for Indian Medicine., 54.
6. Dr. Lohar, Protocol for Testing Ayurvedic, Sidha & Unani medicines, Pharmacopoeial Laboratory for Indian Medicine, Ghaziabad, Department of AYUSH, Ministry of Health & Family Welfare, Government of India.
7. The AyurvedicPharmacopoea of India, Goverment of India, Part-I Vol-VI, Ministry of health & Family Welfare, Department of Health, New Delhi. P: 242, 243, 246, 261, 263. Effective from Ist January 2009.
8. Vikas Gupta, Renu Bansal, Amit Lather and Parveen Bansal "Variation of Heavy Metal Content of Market Variants of Ayurvedic Formulation- Lack of Good Manufacturing Practices (GMP)" *University Centre of Excellence in Research, BFUHS, Faridkot, India. Published 12th April 2013.*