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Physio-Chemical Analysis of Silasathu Parpam

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Abstract

Background: The silasathuparpam is a mineral and salt combination used for treating Urinary Tract Infection. Physiochemical analysis of silasathuparpam was studied for step forward to standardization of drug. **Objective:** To prepare silasathuparpam as per the traditional Siddha literature and to evaluate its chemical changes during drug preparation processes. **Materials and methods:** Karpoora Silasathu, Padikaram, Vengaram, Indhuppu, Vediuppu were purified. ICP-OES was analysed for before and after purification of raw drugs. The purified drugs were powdered separately and mixed well. The mixture was kept in a mud pan, covered by using another mud pan and sealed with clay. It was dried in sunlight and subjected to pudam with fifty cow dung cakes (varaties). Then it was allowed to cool and the final product was collected and ground into fine powder. The particle size of the drug was analysed by using SEM. The physiochemical properties of the drug was evaluated by using ICP-OES and FTIR. **Result :** ICP-OES study of silasathuparpam showed the presence of nutrient elements. The quantity of heavy metals were below the detection level. FTIR study of Silasathuparpam showed a positive deflection to the frequencies of functional groups. SEM study of end product shows that the particle size is of 100 µm. **Conclusion:** Silasathuparpam does not have heavy metals, but contains nutrient elements and a positive deflection to the frequencies of functional groups. Thus the studied physiochemical analysis of silasathuparpam is step forward to standardization of drug.

Keywords: silasathuparpam, siddha, Karpoora Silasathu, Padikaram , Vengaram , Indhuppu, Vediuppu

Introduction

Siddha formulations were either purely herbal or mineral or metal or a combination of herbomineral or metals. Medicines prepared from minerals and salts have long shelf life than herbals and are often used for chronic diseases. The silasathuparpam is a mineral and salt combination used for treating Urinary Tract Infection. Usually preparation of mineral based siddha drug is lengthy procedure and believed that the combinatorial and transmutation changes occur during the process. Literature survey revealed that the

chemical changes during the processing and chemical composition of the drug have not been reported. Thus, the objective of current study to evaluate physiochemical analysis of silasathuparpam. Physiochemical analysis of silasathuparpam was studied for step forward to standardization of drug. The particle size of the drug was analysed by using Scanning Electron Microscope (SEM). The physiochemical properties of the drug was evaluated by using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), Fourier Transform Infrared Spectroscopy (FTIR).

Materials and Methods

The procedures of pre-drug preparation and drug preparation were strictly based on siddha literature. Raw drugs were purchased at Nagercoil, Tamil Nadu, India through proper identification.

Ingredients of silasathuparpam:

Karpoora Silasathu (Selenite)
Padikaram (Alum)
Vengaram (Borax)
Indhuppu (Rocksalt)
Vediuppu (Potassium nitrate)

Purification of Raw Drugs:

1. Karpoorasilasathu:

Karpoorasilasathu was boiled in milk and washed with water then dried

2. Padikaram:

Padikaram was made into fine powder and dissolved in water. The solution was filtered by using a clean cloth and the impurities were removed. The solution was then subjected to heat till it becomes viscous (kulambupatham) and dried.

3. Venkaram:

Venkaram was placed in a mud pan and roasted till the moisture is removed.

4. Indhuppu:

Indhuppu was soaked in vinegar for about three days and then dried in sunlight.

5. Vediuppu:

Sea water were taken in the ratio 2:1 and mixed well. The solution was heated till it boils and dried in sunlight. The process was repeated for about seven times.

Method of preparation:

The purified drugs were taken in the following ratio

Karpoorasilasathu	: 4 varagan (16.4 gm)
Padikaram	: 1 varagan (4.1 gm)
Venkaram	: 1 varagan (4.1 gm)
Indhuppu	: 1 varagan (4.1 gm)
Vediuppu	: 1 varagan (4.1 gm)

The drug were powdered separately and mixed well. The mixture was kept in a mud pan, covered by using

another mud pan and sealed with clay. It was dried in sunlight and subjected to pudam with fifty cow dung cakes (varaties). Then it was allowed to cool and the final product was collected and ground into fine powder. The powder was filtered through a piece of clean cotton cloth.

Adjuvant:

Honey

Indication:

MoothiraKiricharam (Urinary Tract Infection).

Physiochemical analysis:

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM) are done at SAIF, IIT Chennai-36.

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES):

The elemental composition of a sample is often an important part of the information needed to assess its properties. Hence there is a need for sensitive scientific instrumentation like ICP-OES which plays an important role in the determination of these elements. ICP-OES is widely employed for the estimation of metals and metalloids at trace, minor and major concentrations.

Sample Preparation:

About 0.25 gm of test sample and transfer into a liner provided with the instrument. Slowly add 9ml of Nitric acid or Sulphuric acid such that no piece of sample sticks on the slides. Mix thoroughly and allow reacting for few minutes. Place the liner in the vessel jacket. Close the screw cap hand tight in clockwise direction. Seal the vessel and place in the rotor fixed in microwave. Set temperature to 180°C for least 10 minutes. Allow the vessels to cool down to a vessel interior temperature below 60°C and to a vessel surface temperature below 50°C before removing the motor. The digested sample was made upto 100ml with Millipore water. If visible insoluble particles exist, solution could be filtered through whatmann filter paper. Transfer the digested solution into plastic containers and label them properly.

Padikaram (Alum):

Elements	Before Purification	After Purification
Aluminium (Al)	125.783	85.723
Arsenic (As)	BDL	BDL
Boron (B)	-	-
Calcium (Ca)	15.180	11.110
Cadmium (Cd)	BDL	BDL
Mercury (Hg)	BDL	BDL
Iron (Fe)	1.540	1.240
Potassium (K)	810.215	520.205
Sodium (Na)	1.854	1.654
Phosphorus (P)	19.280	15.200
Lead (Pb)	BDL	BDL
Sulphur (S)	211.252	151.202
Selenium (Se)	-	-

Indhuppu (Rock salt):

Elements	Before Purification	After Purification
Aluminium (Al)	-	-
Arsenic (As)	-	-
Boron (B)	-	-
Calcium (Ca)	15.180	10.110
Cadmium (Cd)	BDL	BDL
Mercury (Hg)	BDL	BDL
Iron (Fe)	1.590	1.110
Potassium (K)	10.215	10.015
Sodium (Na)	741.854	689.104
Phosphorus (P)	19.280	15.140
Lead (Pb)	BDL	BDL
Sulphur (S)	1.252	1.002
Selenium (Se)	-	-

Vediuppu (Potassium nitrate):

Elements	Before Purification	After Purification
Aluminium (Al)	-	-
Arsenic (As)	BDL	BDL
Boron (B)	-	-
Calcium (Ca)	16.253	12.860
Cadmium (Cd)	BDL	BDL
Mercury (Hg)	BDL	BDL
Iron (Fe)	1.562	1.241
Potassium (K)	815.23	786.08
Sodium (Na)	1.135	1.003
Phosphorus (P)	19.185	13.685
Lead (Pb)	-	-
Sulphur (S)	1.856	1.258
Selenium (Se)	-	-

Vengaram (Borax):

Elements	Before Purification	After Purification
Aluminium (Al)	-	-
Arsenic (As)	BDL	BDL
Boron (B)	235.215	180.241
Calcium (Ca)	40.225	30.22
Cadmium (Cd)	BDL	BDL
Mercury (Hg)	-	-
Iron (Fe)	1.88	1.56
Potassium (K)	95.33	45.52
Sodium (Na)	283.35	222.856
Phosphorus (P)	25.29	20.98
Lead (Pb)	-	-
Sulphur (S)	20.112	0.457
Selenium (Se)	-	-

Karpooora silasathu (Selenite):

Elements	Before Purification	After Purification
Aluminium (Al)	-	-
Arsenic (As)	BDL	BDL
Boron (B)	-	-
Calcium (Ca)	215.180	204.32
Cadmium (Cd)	BDL	BDL
Mercury (Hg)	BDL	BDL
Iron (Fe)	1.140	1.100
Potassium (K)	20.215	15.355
Sodium (Na)	5.854	3.224
Phosphorus (P)	19.280	14.320
Lead (Pb)	BDL	BDL
Sulphur (S)	21.252	11.252
Selenium (Se)	228.241	206.11

Silasathu parpam:

Elements	SilasathuParpam
Aluminium (Al)	25.783
Arsenic (As)	BDL
Boron (B)	50.24
Calcium (Ca)	15.180
Cadmium (Cd)	BDL
Mercury (Hg)	BDL
Iron (Fe)	1.040
Potassium (K)	30.215
Sodium (Na)	15.214
Phosphorus (P)	14.180
Lead (Pb)	BDL
Sulphur (S)	1.250
Selenium (Se)	48.201

Purified Karpoora Silasathu shows reduction in Calcium (Ca), Iron (Fe), Potassium (K), Sodium (Na), Phosphorus (P), Sulphur (S), Selenium (Se) than before purification. Purified Padikaram shows reduction in Aluminium (Al), Calcium (Ca), Iron (Fe), Potassium (K), Sodium (Na), Phosphorus (P), Sulphur (S) than before purification. Purified Indhuppu shows reduction in Calcium (Ca), Iron (Fe), Potassium (K), Sodium (Na), Phosphorus (P), Sulphur (S) than before purification. Purified Vediuppu shows reduction in Calcium (Ca), Iron (Fe), Potassium (K), Sodium (Na), Phosphorus (P), Sulphur (S) than before purification. Purified venkaram shows reduction in Boron (B), Calcium (Ca), Iron (Fe), Potassium (K), Sodium (Na), Phosphorus (P), Sulphur (S) than before purification.

At the end of product Silasathuparpam shows reduction in Aluminium (Al), Boron (B), Calcium (Ca), Iron (Fe), Potassium (K), Sodium (Na), Phosphorus (P), Sulphur (S), Selenium (Se) than before purification. Arsenic (As), Cadmium (Cd), Mercury (Hg), Lead (Pb), are shows below detection level.

Fourier Transform Infrared Spectroscopy (FTIR):

Vibrational spectroscopy is an extremely useful tool in the elucidations of molecular structure. The spectral bands can be assigned to different vibrational modes of the molecule. The various functional groups present in the molecule can be assigned by a comparison of the spectra with characteristic functional group frequencies. As the positions of the bands are directly related to the strength of the chemical bond, a large number of investigations including intermolecular interactions, phase transitions and chemical kinetics can be carried out using this branch of spectroscopy.

Table of characteristics IR absorption:

Frequency, cm ⁻¹	Functional group	Result
3640 – 3610 (s,sh)	Free hydroxylalcohols, phenols	-
3500 – 3200 (s,b)	Alcohols, phenols	-
3400 – 3250 (m)	Primary, secondary amines and amides	-
3300 – 2500 (m)	Carboxylic acids	-
3330 – 3270 (n,s)	Alkynes(terminal)	-
3100 – 3000 (s)	Aromatics	+
3100 – 3000 (m)	Alkenes	+
3000 – 2850 (m)	Alkanes	+
2830 – 2695 (m)	Aldehydes	-
2260 – 2210 (v)	Nitriles	-
2260 – 2100 (w)	Alkynes	-
1760 – 1665 (s)	Carbonyls (general)	+
1760 – 1690 (s)	Carboxylic acids	+
1750 – 1735 (s)	Esters, saturated aliphatic	-
1740 – 1720 (s)	Aldehydes, saturated aliphatic	-
1730 – 1715 (s)	Alpha,bête unsaturated esters	-
1715 – 1710 (s)	Ketones,saturated aliphatic	-
1710 – 1665 (s)	Alpha,beta-unsaturated aldehydes,	+

In IR spectroscopy, the resonance absorption is made possible by the change in dipole moment accompanying the vibrational transition. The Infrared spectrum originates from the vibrational motion of the molecule. The vibrational frequencies are a kind of fingerprint of the compounds. This property is used for characterization of organic, inorganic and biological compounds. The band intensities are proportional to the concentration of the compound and hence qualitative estimations are possible.

The IR spectroscopy is carried out by using Fourier transform technique.

Sampling techniques:

There are variety of techniques for sample preparation depending on the physical form of the sample to be analysed.

Solid	:	KBr or Nujol mull method.
Liquid	:	CsI / TlBr Cells
Gas	:	Gas cells

Experimental Procedure:

Done at SAIF,IIT Chennai-36.
KBr Method

The sample was grounded using an agate mortar and pestle to give a very fine powder. The finely powder sample was mixed with about 100mg dried KBr salt. The mixture was then pressed under hydraulic press using a die to yield a transparent disc (measure about 13mm diameter and 0.3mm in thickness), through which the beam of spectrometer passed.

	ketones	
1680 – 1640 (m)	Alkenes	-
1650 – 1580 (m)	Primary amines	+
1600 – 1585 (m)	Aromatics	-
1550 – 1475 (s)	Nitro compounds	+
1500 – 1400 (m)	Aromatics	+
1470 – 1450 (m)	Alkanes	-
1370 – 1350 (m)	Alkanes	-
1360 – 1290 (m)	Nitro compounds	+
1335 – 1250 (s)	Aromatic amines	+
1320 – 1000 (s)	Alcohols, carboxylic acids, esters, ethers	+
1300 – 1150 (m)	Alkyl halides	+
1250 – 1020 (m)	Aliphatic amines	+
1000 – 650 (s)	Alkenes	+
950 – 910 (m)	Carboxylic acids	-
910 – 665 (s,b)	Primary secondary amines	+
900 – 675 (s)	Aromatics	+
850 – 550 (m)	Alkyl halides	+
725 – 720 (m)	Alkanes	+
700 – 610 (b,s)	Alkynes	+
690 – 515 (m)	Alkyl halides	+

m= medium, w=weak, s=strong, n=narrow, b=broad, sh=sharp

Table shows the presence of functional groups such as aromatics, alkenes, alkanes, carbonyls, carboxylic acids, alpha and beta unsaturated aldehydes, ketones, nitro compounds, aromatic amines, alcohols, aliphatic amines, primary amines, secondary amines, alkyne alkyl halides.

Scanned Electron Microscopy (SEM)

A SEM is essentially a high magnification microscope, which uses a focussed scanned electron beam to produce images of the sample, both top - down and, with the necessary sample preparation, cross-sections. The primary electron beam interacts with the sample in a number of key ways:-

Primary electrons generate low energy secondary electrons, which tend to emphasize the topographic nature of the Specimen.

Primary electrons can be backscattered which produces images with a high degree of atomic number (Z) contrast.

Ionized atoms can relax by electron shell-to-shell transitions, which lead to either X-ray emission or Auger electron ejection. The X-rays emitted are characteristic of the elements in the top few μm of the sample.

The SEM is carried out by using FEI-Quanta FEG 200-High Resolution Instrument.

Resolution: 1.2 nm gold particle separation on a carbon substrate

Magnification: From a min of 12x to greater than 1, 00,000 X

Application: To evaluate grain size, particle size distributions, material homogeneity and inter metallic distributions.

Experimental Procedure:

Done at SAIF, IIT Chennai-36.

Sample preparation:

Sample preparation can be minimal or elaborate for SEM analysis, depending on the nature of the samples and the data required. Minimal preparation includes acquisition of a sample that will fit into the SEM chamber and some accommodation to prevent charge build-up on electrically insulating samples. Most electrically insulating samples are coated with a thin layer of conducting material, commonly carbon, gold, or some other metal or alloy. The choice of material for conductive coatings depends on the data to be acquired: carbon is most desirable if elemental analysis is a priority, while metal coatings are most effective for high resolution electron imaging applications. Alternatively, an electrically insulating sample can be examined without a conductive coating in an instrument capable of "low vacuum" operation.

Results

The particles were stabilized and have irregular morphology. The particles were distributed in μ range and the size is 100 μm .



Discussion

The ingredients of Silasathuparpam were purified and the drug was prepared according to the process mentioned in the text sarabendhirarsoolai, moola, kusta, pitharogasikitchaimuraigal Pg.No: 174. This drug is mainly used in treating the diseases of urinary system.

ICP-OES study indicated the presence of Calcium, Iron, Potassium, Sodium, Phosphorus and the heavy metals contained in the raw drug samples were below the detection level.

ICP-OES study of the purified ingredient samples revealed a considerable decrease in the quantity of Aluminium, Potassium, Phosphorus, Sulphur, Sodium, Selenium.

ICP-OES study of silasathuparpam showed the presence of nutrient elements. The quantity of heavy metals were below the detection level.

FTIR study of Silasathuparpam showed a positive deflection to the frequencies corresponding to the following functional groups. aromatics, alkenes, alkanes, carbonyls, carboxylic acids, alpha and beta unsaturated aldehydes, ketones, nitro compounds,

aromatic amines, alcohols, aliphatic amines, primary amines, secondary amines, alkyne alkyl halides.

SEM study of end product shows that the particle size is of 100 μm .

Conclusion

ICP-OES study of silasathuparpam showed the presence of nutrient elements. The quantity of heavy metals were below the detection level. FTIR study of Silasathuparpam showed a positive deflection to the frequencies of functional groups. SEM study of end product shows that the particle size is of 100 μm . Thus the studied to evaluate physiochemical analysis of silasathuparpam is step forward to standardization of drug.

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