Research Paper

Physico-Chemical Standardisation and Instrumental Analysis of Gandhaga Parpam A Poly Herbo Mineral Siddha Formulation

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ABSTRACT
Siddha system is a treasure house of a secret science embodying several medicines for curing several diseases. But this is a time to standardise siddha drugs for world acceptance for the safety concerned. This paper deals with the physico-chemical properties, and Instrumental analysis of Gandhaga parpam for part of standardisation as per AYUSH Guidelines. The physico-chemical analysis the drug shows the quality of the drug. FTIR Interprets the molecular structure of the sample. SEM analysis showed the size of the drug particle, is in Nanometre which denotes that the trial drug could have potent drug delivery. XRF analysis disclosed the percentage of elements presence in the drug. Based on the results the drug Gandhaga parpam was standardised as part. Further advanced study will need to full fill the standardisation. This paper will help the physicians and researchers to do the needful in the world health society.

Keywords: Gandhaga parpam, Siddha, physico-chemical analysis, AYUSH Guidelines, Instrumental analysis.

INTRODUCTION
Siddha system is a treasure house of a secret science embodying several medicines for curing several diseases. In Siddha system has its own well developed chemistry and the Siddhars training in the direction of its development had resulted in the genesis of thousand of minerals and metallic preparations, thus the system is well equipped to compact any type of diseases. But this is a time to standardise siddha drugs for world acceptance for the safety concerned.

World Health Organization (WHO) has appreciated the importance of medicinal plants for public health care. The process of evaluating the quality and purity of herbo mineral drugs by means of various parameters like physical, chemical and biological observation is called standardization.

One of the poly herbo mineral siddha drug Gandhaga parpam (GP) indicated for several types of arthritis1. This paper deals with the physico-chemical properties and Instrumental analysis of Gandhaga parpam for part of standardisation as per AYUSH Guidelines.

MATERIALS AND METHODS
Preparation of Gandhaga parpam:
Drug selection:
Gandhaga parpam was taken as a compound drug for several arthritis from the literature of...
Ingredients of the test drug:
Gandhagam (sulphur), Elumichai (lemon), and Maruthampattai ash (Terminalia arjuna)

Collection and Authentication of Raw Drugs
The drug Gandhagam and Maruthampattai were bought from country raw drug shop at parry’s corner Chennai, Tamilnadu. Lemon bought from Koyembedu market. The sulphur was identified and authenticated by the experts of Gunapadam (Pharmacology) department, Govt. Siddha Medical College, Arumbakkam, Chennai-106. The lemon and maruthampattai was identified and authenticated by the Gunapadam experts. Govt. Siddha Medical College, Arumbakkam Chennai -106. After identification, the samples of raw materials had been conserved in the laboratory of the department for future reference.

Preparation of the drug:
Parpam is one of the higher medicines of the Siddha Medical System. Gandhaga Parpam is made from the Mineral sulphur. The materials required for the processing of Gandhaga Parpam to convert it as the end product is as follows.

Purification of Gandhagam (sulphur):
Gandhagam was soaked in lemon juice for 24 hours.

Materials Required:
1. Purified Sulphur (Gandhagam) – 1 palam (35 g)
2. Bark of Maruthampattai – 3 Kg
3. Lemon -20
4. Earthen vessel
5. Kada cloth
6. No. of cow dung cakes – 100 palam (3.5 kg)

Preparation of Maruthampattai ash:
The Bark of Maruthampattai (Terminalia arjuna) was burnt and ash was collected.

Preparation of parpam and storage:
Half of the earthen vessel was filled with maruthampattai ash and 35 gm of the purified Gandhagam was placed in the middle of the ash. Then it placed in an earthen crucible, it was sealed with a mud pasted cloth seven times. Then it dried under sunlight for one day.

Incineration and trituration:
Then the earthen crucible was subjected to pudam (incineration) process with cow dung cakes of weight of 100 palam (3.5 kg). After finishing pudam process let the earthen crucible undisturbed to give away heat. The seal was opened on the next day. The sulphur was collected and grounded well. Finally parpam is prepared. The colour of the parpam is yellowish white colour and the consistency was very fine.

Storage:
The drug Gandhaga parpam was stored in a clean air tight glass container for following analysis.

Standardization as per siddha classical literature:
The drug Gandhaga parpam was examined by some basic techniques mentioned in Siddha literatures as follows

1. Colour: The Gandhaga parpam was taken in a Petri dish and observed the colour.
2. Floating on Water: A pinch of Gandhaga parpam was sprinkled over the water in a glass container.
3. Finger Print Test: A pinch of Gandhaga parpam was taken and rubbed in between the thumb and index finger.
4. Lustre: The Gandhaga parpam was taken in a Petri dish and observed for any lustre in daylight via magnifying glass.
5. Taste: Small amount of parpam was kept in the tip of the tongue.

Standardization of Gandhaga parpam by using modern techniques:
Standardization of drug helps to authenticate and determine its quality and efficiency. Standardization of herbo mineral drug is rooted in qualitative and quantitative analysis by means of physico-chemical properties and instrumental analysis.

Physico Chemical Parameters:
Loss on drying at 105°C, total ash, water soluble ash, acid-insoluble ash, were carried out as per the procedures mentioned in standard reference.
Physical characterization:
Colour, state of drug, consistency, solubility, sense on touch, sense on taste, sense on smell, pH value, Action on heat, Flame test, Ash test were carried out.

Bio-Chemical analysis:
Qualitative test for various acid and basic radicals were carried out as per the methods mentioned in standard practical guide.

Instrumental analysis:
The chemical finger print was engaged by using modern analytical technique Fourier Transform Infra-Red Spectroscopy (FTIR).  

Instrument details
Model : Spectrum one: FT-IR Spectrometer
Scan Range : MIR 450-4000 cm-1
Resolution : 1.0 cm-1
Sample required : 50 mg, solid or liquid

The quantitative and qualitative analysis of chemical elements was carried out by using X-Ray Fluorescence Spectroscopy (XRF).

Make: Bruker ,
Model : S4 PIONEER

The particle size and qualitative analysis of chemical elements of *Gandhagarparpam* were also assessed by Scanning Electron Microscope (SEM).

Instrument details
Model : SEM-Hitachi
Scan Range : S-3400n
Resolution : 1.2 nm gold particle separation on a carbon substrate
Magnification : From a min of 12 x to greater than 1, 00,000 X

RESULTS AND DISCUSSION
Standardisation as pre siddha literature:

1. Colour: The word *parpam* means white and fine. Mostly *parpam* are in white and ivory colour. *Gandhaga parpam* was in yellowish white or ivory in colour. It shows the perfect colour of parpam.

2. Floating on Water: The *parpam* particles did not sink but floated on the water surface. It indicates the lightness of *Gandhaga parpam*.

3. Finger Print Test: It entered into the lines of the fingers. It confirmed the fineness of parpam.

4. Lustre: The drug did not show any glowing particles, it shows that the drug is not possess unchanged substances like metals and other toxic substances. So, the drug was properly prepared.

5. Taste: Tasteless and felt mild irritation, due to its nature of sulphur.

| S.NO | Parameter              | Result                                      |
|------|------------------------|---------------------------------------------|
| 1    | Colour                 | Yellowish White colour                      |
| 2    | State of the drug      | Powder                                      |
| 3    | Consistency            | Fine powder                                 |
| 4    | Solubility             | Sparingly soluble in water. Well soluble in acids (Hcl and H2SO4) |
| 5    | Sense on touch         | Fine                                        |
| 6    | Sense on taste         | Tasteless. Slight irritation of the medicine. When tasted with adjuvant there is no irritation felt. |
| 7    | Sense of smell         | Mild rotten egg smell                       |

Specific gravity of *Gandhaga parpam* was less than the water. Thus, it indicates the lightness of the drug.

pH of the trial drug was. It shows mild irritation of the drug. The adjuvant of the drug is honey, butter, ghee, which reduce that irritation and bad odour. Hence, this adjuvant reduces the acidity of *Gandhaga parpam*. According to pharmacokinetics, mild acidic drugs are absorbed in acidic environment that is in the stomach.
Particle size analysis indicates *Gandhaga parpam* contain very fine particles. The mesh aperture for sieve size no: 120 is 125µm.

### Determination of Ash Values

#### Percentage total Ash

| Weight of the drug powder taken (gm) | Weight of Ash obtained (gm) | Percentage w/w total ash | Mean value |
|-------------------------------------|-----------------------------|--------------------------|------------|
| 3.0                                 | 2.94                        | 98                       |            |
| 3.0                                 | 2.93                        | 97.6                     |            |
| 3.0                                 | 2.90                        | 96.6                     | 97.4%      |

#### Percentage water soluble ash values

| Weight of the drug powder taken (gm) | Weight of Ash obtained (gm) | Percentage w/w total ash | Mean value |
|-------------------------------------|-----------------------------|--------------------------|------------|
| 3.0                                 | 0.293                       | 9.76                     |            |
| 3.0                                 | 0.292                       | 9.73                     |            |
| 3.0                                 | 0.294                       | 9.8                      | 9.76%      |

#### Percentage acid insoluble ash values

| Weight of Sample taken (gms) | Weight of acid insoluble ash obtained (gm) | Percentage w/w acid insoluble ash | Mean value |
|-----------------------------|--------------------------------------------|----------------------------------|------------|
| 3.0                         | 0.013                                      | 0.43                             | 0.38%      |
| 3.0                         | 0.012                                      | 0.4                               |            |
| 3.0                         | 0.010                                      | 0.33                              |            |

### Determination of Loss on Drying

#### Table 10: Percentage loss in weight on drying

| Weight of the powder taken (gms) | % Loss in weight (w/w) | Mean value |
|----------------------------------|------------------------|------------|
| 1.5                              | 0.15                   |            |
| 1.5                              | 0.13                   |            |
| 1.5                              | 0.16                   | 0.146%     |

From the result of preliminary chemical analysis reveals that trial drug *Gandhaga parpam* has sulphate, chloride, phosphate, calcium, potassium, magnesium, iron.

### FTIR

The results of Table no:14 and Fig no:9 shows the presence of functional group and inorganic compounds of *Ghandhaga parpam*.

FT-IR is a very useful tool in the recognition of the functional groups of bio molecules, thus aiding in their structural elucidation, so confirming the presence of active molecules responsible for the therapeutic activity of Siddha drugs.

### Interpretation

This table shows that total ash value was found to be very high due to the presence of high organic matter.

Physico-chemical analysis of *Ghandhaga parpam* showed that Loss on drying (LOD) 0.146% which shows that low moisture content present in the prepared medicine. Increased moisture content is the issue for instability of a drug and lesser shelf life of a drug. Since *Ghandhaga parpam* was well prepared, it could get maximum stability and better shelf life. Longer shelf life 100 years for *parpam* mentioned in Siddha literature is justified from the above observation.

### Preliminary Basic and Acidic Radical Studies

#### Results of basic and acidic radical studies

| S.no  | Parameter           | Result |
|-------|---------------------|--------|
| 1.    | Test for Potassium  | +      |
| 2.    | Test for Calcium    | +      |
| 3.    | Test For Magnesium  | +      |
| 4.    | Test For Ammonium   | -      |
| 5.    | Test For Sodium     | +      |
| 6.    | Test for Iron (Ferrous) | +  |
| 7.    | Test For Zinc       | -      |
| 8.    | Test For Aluminium  | -      |
| 9.    | Test For Lead       | -      |
| 10.   | Test for Copper     | -      |
| 11.   | Test For Mercury    | -      |
| 12.   | Test for Arsenic    | -      |
| 13.   | Test for Sulphate   | +      |
| 14.   | Test for Chloride   | +      |
| 15.   | Test for Phosphate  | +      |
| 16.   | Test for Carbonate  | -      |
| 17.   | Test for fluoride &oxalate | - |
| 18.   | Test For Nitrate    | -      |
Interpretation of FTIR Spectrum

| Wave number(cm⁻¹) | Vibrational modes of Gandhagaparppam in IR region |
|------------------|--------------------------------------------------|
| 3879.6           | O-H Stretching                                   |
| 3788.5           | O-H Stretching                                   |
| 3409.5           | O-H Bending                                      |
| 2937.8           | C-H Bending                                      |
| 2365.5           | C=O Stretching                                   |
| 2354.2           | C=O Stretching                                   |
| 2343.3           | C=O Stretching                                   |
| 1613.4           | C=C Bending                                      |
| 1409.6           | C-C Bending                                      |
| 1051.8           | C-C Bending                                      |
| 816.9            | C-O Bending                                      |
| 782.5            | C-O Bending                                      |
| 668.7            | C-O Bending                                      |
| 488.0            | C-O Bending                                      |
| 464.2            | C-O Stretching                                   |

XRF (X-RAY Fluorescence spectroscopy):
X-ray fluorescence is used to determine the chemical elements both qualitatively and quantitatively by measuring their characteristic radiation of the sample.

XRF result of Gandhaga parpam

Element in oxide form

| Formula | Concentration (%) |
|---------|-------------------|
| SO₃     | 98.73             |
| Cl      | 0.43              |
| Na₂O    | 0.36              |
| K₂O     | 0.12              |
| SiO₂    | 0.09              |
| MgO     | 0.07              |
| P₂O₅    | 0.06              |
| Fe₂O₃   | 0.05              |
| Al₂O₃   | 0.04              |
| CuO     | 0.04              |
| CuO₃    | 0.01              |
| Pd      | 50ppm             |
| MoO₃    | 34ppm             |

Element form

| Formula | Concentration (%) |
|---------|-------------------|
| S       | 99.10             |
| Cl      | 0.43              |
| Na      | 0.27              |
| K       | 0.10              |
| Mg      | 0.04              |
| Si      | 0.04              |
| Fe      | 0.04              |
| Ca      | 0.03              |
| P       | 0.02              |
| Al      | 0.02              |
| Cu      | 92ppm             |
| Pd      | 60ppm             |
| Mo      | 23ppm             |
XRF result shows the presence of sulphur tri oxide in 98.7% .Mainly Gandhaga parpam is contains oxide form of elements. It is due to the calcined process.

**SEM (Scanning Electron Microscope with Energy Dispersive X-Ray Analysis):**
The SEM picture of *Gandhaga parpam* is shown in Fig no:8 & 9.

![SEM Picture of Gandhaga parpam](image)

SEM picture shows nano particles in *Gandhaga parpam*

SEM picture shows nanoparticle and micro particle size of the sample. The particle size varies between 60 nm to 172 nm. The extremely small size of nanoparticles allows them to penetrate cells and interact with cellular molecules. Nanoparticles have significant properties that can be used to enhance drug delivery. As the particle is in nano size, a low dose of the drug is enough to treat diseases. Hence *Gandhaga parpam* which is prepared biologically contains nanoparticles to enhance fast pharmacological action in target site.

**CONCLUSION**
Various analysis such as physicochemical, biochemical analysis, instrumental analysis were made. From the above analysis we came to know the presence of active ingredients responsible for its activity. Biochemical analysis showed the presence of sodium, magnesium, potassium, calcium and sulphate. Thus from these results we come to know the effectiveness of the drug is due to the presence of these constituents and it has a synergistic effect in acting against the disease.

The instrumental analysis FTIR showed the peak values present which are the functional groups responsible for its activity. XRF result shows the presence of sulphur tri oxide in 98.7%.SEM picture described its morphology and the particle size. Mainly *Gandhaga parpam* is contains oxide form of elements. It is due to the calcined process. Based on the results the drug *Gandhaga parpam* was standardised as part. Further advanced study will need to full fill the standardisation. This paper will help the physicians and researchers to do the needful in the world health society.

**REFERENCE**
1. Abdhula Sayubu PM. Anubhoga Vaidhya Navaneetham Part I. Thamarainoolagam, Chennai (2006) 106.pg:28
2. Quality control standards of certain formulations, Published by CCRAS, New Delhi, 1991, pg 78..
3. Anonymous .Quality Control Methods for Medicinal Plant Materials. World Health Organisation, Geneva, Switzerland.1998.
4. Lohar DR. Pharmacopoeial laboratory for Indian medicine. Dept of Ayurvedha, yoga and Naturopathy, Siddha, Unani and Homoeopathy (AYUSH), Ministry of health and family welfare. Newdelhi. 2011; 20.
5. Geochemical instrumentation and analysis Ava online at http://serc.carleton.edu/research_education/geochemsheets/index.html
6. Scanning electron microscope Available online at http://www.mee-inc.com/hamm/scanning-electron-microscopy-sem/