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Physicochemical Characterization and Instrumental Analysis of the Siddha Mineral Drug Naga Parpam

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ABSTRACT

The aim of the present study was to standardize the physico-chemical traits of the Naga Parpam, a Siddha traditional drug for treating oligospermia so as to attain maximum benefit to the mankind. The organoleptic characters, physico-chemical characters like ash values, pH value, specific gravity, solubility were analyzed. The total ash value was found to be 27.2% w/w, acid insoluble ash value is 11.6% w/w and loss of drying at 105 ° c is 0.29 % w/w. The pH value is 9.6. The SEM analysis of the sample showed the presence of nano and. The quantitative analysis of the sample through EDAX revealed the content of Zinc, Carbon, Oxygen, Potassium, Iron, Chloride, Potassium, Calcium. The FT-IR spectroscopy applied in the mid infra-red region 4000 cm⁻¹ to 400 cm⁻¹ revealed the presence of functional groups like primary aliphatic amines, alcohols, alkynes, esters, carboxylic acids. The XRF of Naga Parpam revealed the elements like zinc, potassium, sulphur in oxide form as well as in elemental form. This study highlights the suitable application of modern standardizing techniques for bringing the herbal formulation into focus.

Keywords: Physico-chemical traits, ash values, SEM, FT-IR spectroscopy

ARTICLE INFO

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1. Introduction

The Siddha System of medicine is an ancient traditional system of medicine signifying uniqueness in drug preparations with effective therapeutic outcomes and minimal or no side effects. In the recent years a new breakthrough has occurred in the medical field over the usage of herbals and minerals and it is quite evident when we check the usage of herbals for many health hazards. This prevalent usage is increasing to a tremendous extent in this modern era.

The World Health organization defines herbal medicines as those containing plant parts or plant materials in raw state or processed form containing active principles [1]. Siddha system possesses bountiful forms of herbal, herbo-mineral preparations in which many combinations, new formulations are developed and practiced every now and then. Amongst the herbo-mineral preparations, Parpam is a calcined medicinal form obtained due to repeated incinerations of purified metal along with juices of

medicinal plants. Naga parpam, the drug considered in this present study comes under the parpam category is an effective medicine for treating oligospermia. During the preparation of this medicine, adulteration of the drugs and misidentification of the herbs are possible which may lead to serious effects and problems in pharmaceutical industry. Thus it is necessary to produce high quality formulations without any adverse reactions.

To overcome all these difficulties and to get a better quality of medicines a standard and universally accepted drug standardisation for each drug should be made mandatory. For this the WHO and AYUSH have suggested certain guidelines to be followed during the standardisation procedures. In this study an attempt was made to furnish organoleptic characters, physico-chemical analysis, biochemical analysis and instrumental analysis with a notion of standardising the drug Naga parpam in such a way this would be a further reference for any analytical studies.

2. Materials and Methods

Drug selection:

The drug Naga Parpam (Zincum) was prepared in steps according to the procedures mentioned in the classical Siddha Literatures [2]. The main ingredients included in the formulation are Nagam (Zinc) and Palasu (Butea monosperma). The raw drugs were collected from the hills of Vellore district and Zinc was procured from the country raw drug shop at Parrys Chennai. The drugs were identified and authenticated by the experts of Pharmacognosy Department of Siddha Central Research Institute, Chennai and faculties of Department of Pharmacology, Govt. Siddha Medical College, Chennai. The specimen sample of each ingredient was labeled individually and kept in the department for future reference.

Purification and Preparation of the drug:

Zinc, the main ingredient of Naga parpam was purified by removing its toxic ingredients by the process described below. The ghee of south Indian Mahua (*Madhuca longifolia*)-Iluppai ghee was taken in a mud pot. Two crystallized pieces of Ammonium chloride were placed in a pot in such a way that half of the portion of the pieces was immersed in the ghee in opposite direction. The melted zinc in an iron pot was poured into the ghee of south Indian mahua. After that, the melted Zinc was allowed to cool. Then the process was repeated for twenty another times and the purification was completed.

Preparation of Purasam Poo Kuzhi Tailam

The primary step in the preparation of Naga Parpam was the extraction of kuzhi tailam. Kuzhi tailam refers to a type of oil extracted from a special set up called Kuzhi thaila karuvi. Flowers of Mahua weighing 3 kg were placed inside

the multiple-holed earthen pot and its lid was closed by another earthen lid with the mud pasted cloth. A pit was dug out and the ceramic container was placed in the pit. Then, the earthen pot was placed on the ceramic bowl as such the holes facing towards the opening of the bowl and it was tightly sealed with the mud pasted cloth. The exterior of the ceramic bowl was tightly packed with sand and the space around the earthen pot was surrounded with cow dung cakes and ignited. When the ignition is completed, the setup was allowed to cool. Finally, the oil produced from the flowers of *Butea monosperma* was collected in the container. The oil obtained from this special process is called the Purasam poo kuzhi tailam. 3 kg of flowers yielded 700 ml of oil.

Preparation of parpam and storage:

35 gm of zinc was melted in a large based spoon and then Purasam poo kuzhitailam was slowly poured. The process was repeated several times until the zinc was transformed into colourless flakes called Naga Parpam. The final drug was collected and preserved in an airtight container and labeled as NP.

Siddha analytical parameters for parpam

The parameters specific to parpam were carried out as per the guidelines mentioned in the Siddha classical texts [3]. A pinch of parpam when kept on the tongue should be tasteless and it should float on the water. Moreover parpam when placed between the thumb and index finger should enter the furrows of the finger. The thumb when pressed on the parpam shows a clear fingerprint. The parpam should not possess any lustre on seeing through naked eye.

Physico-chemical analysis of the drug

Naga Parpam was subjected for the determination of organoleptic characters like colour, odour, taste, texture, hardness and physicochemical parameters such as total ash,

3. Instrumental Analysis

Scanning Electron Microscope Analysis (SEM)

SEM analysis [4] was done mainly to study the morphology of the grains and the crystalline structure and arrangement of the particles. It was carried out at a magnification range of 12 X to 1,00,000X using S-3400n SEM-Hitachi at Anna University, Chennai. A focussed beam of high energy electrons is allowed to pass to generate signals at the surface of the sample drug. The signals derived from the interactions between the sample and electrons produce an image on the screen showing the details including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample.

Energy Dispersive X-ray Analysis (EDAX)

The SEM instrument equipped with EDAX enabled the instrument to perform compositional analysis of the sample Naga parpam.

Fourier Transform–Infra Red Spectroscopy Study (FTIR)

3. Results and Discussion

The results of Siddha testing parameters are shown in the table 1

Table 1: Siddha analytical parameters of parpam

S.No	Siddha parameters	Inference
1	Colour	White
2	Lustre	Lustreless
3	Fingerprint Examination	Entered into the lines of the finger
4	Floatability	Floats on the surface of water
5	Taste	Tasteless

The total ash was found to be 27.2% indicating a less amount of organic matter [7] and a significant amount of minerals. The drug possesses a low value (11.6 %) of acid insoluble ash indicating that the preparation did not contain any siliceous matter [8] and the medicine was prepared in a hygienic condition signifying a better quality of drugs. The loss on drying test at 105°C indicates that only of 0.29 % of water and volatile component have been lost when 1g of NP kept at 105°C. This moisture content prevents reduction of efficacy and degeneration. So the shelf life has been dated up to 75 years as mentioned in classical Siddha literature. The pH value at 25°C was found to be 9.6 which indicates the alkalinity of the drug.[9] The size of the particle is reduced to nano particles[10] which passes through sieve no: 44 indicating the fineness so that the drug is easily assimilable in the digestive system.

acid insoluble ash, water soluble ash, moisture content, alcohol and water soluble extractives, loss of weight at 105° C were evaluated according to the standard procedures.

The IR data was acquired with Spectrum one FT-IR Spectrometer at Anna University, Chennai adopting the KBr Pellet procedure [5]. In this method 0.25-0.50 teaspoons of KBr was mixed thoroughly mixed with solid powder of Naga parpam and ground finely in an agate mortar with the pestle until it became very fine. The sample was made into a pellet by placing in a pellet die and pressed at 5000-10,000 psi placed in the FTIR sample holder. The computer was turned on and the software launched provide spectrum of graphs with peaks and the results were printed.

X-Ray Fluorescent Analysis

XRF analysis [6] was carried out at Sastra University, Tanjore using Bruker S8 Tiger XRF Spectrometer. The drug sample of about Naga parpam was taken in aluminium cups and 2 grams of boric acid was placed in the cup and the sample was topped with boric acid again. A high pressure of about 25 tonnes was applied using hydraulic press to pelletise the aluminium cups to 34 mm pellets. The results were obtained and were tabulated below.

The results of the organoleptic characters are shown in the table 2. as follows,s

Table 2: Organoleptic characters of Naga Parpam

S.No	Parameter	Results
1	Loss on Drying at 105°C	0.29 %
2	Total Ash	27.2 %
3	Acid insoluble Ash	11.6 %
4	Particle size	Completely passes through sieve no.44
5	pH	9.6

Scanning Electron Microscopy (SEM) Analysis

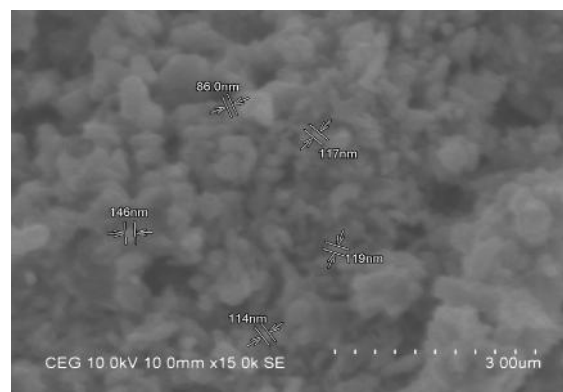


Figure 1: Showing the picture of SEM

The particles of Naga Parpam are in Ultra Nano size of 86.0 nm. The other random particles showed the nano particles of the size 114nm, 117nm, 119nm and 146nm. Nano particles of metal oxides such as zinc oxide have attracted increasing technology interest because they are bio safe and

bio-compatible and can be used for bio medical applications without coating and toxicity. With these unique characteristics, Zinc oxide is one of the most important nano materials in future research and applications [11s].

Energy Dispersive X-Ray Analysis (EDAX):

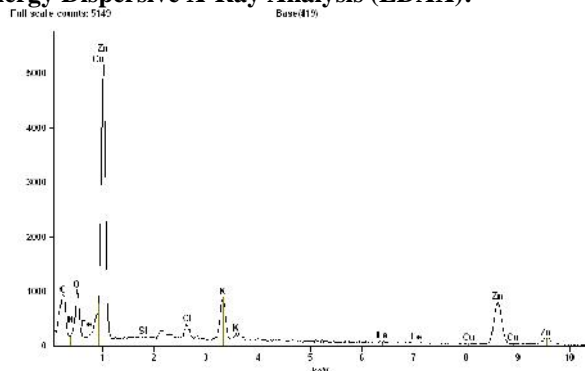


Figure 2: Showing the graph of EDAX analysis

These trace quantities of minerals quantitatively assessed by EDAX analysis along with carbon, oxygen may play an important role in the functioning of various enzymes in biological systems and have immunomodulatory [12] functions and thus influence the susceptibility to the course and the outcome of a variety of viral infections. Zinc element is directly involved in the production, maturation and activation of sperm. It has also antibacterial property^[13]. Calcium is responsible for the bone remodeling and serves as a main storage material in bones.^[14] These elements are present in minimal quantities and heavy metals

The EDAX results showed the presence of the following elements and it is shown in the table.2. as follows,

Table 3: Showing the results of EDAX analysis

Element	Net weight	Weight %	Atom %
C	5342	23.43	52.70
N	465	1.96	3.77
O	8291	8.58	14.50
Cl	3538	1.36	1.04
K	9862	4.27	2.95
Fe	315	0.48	0.28
Cu	45	0.12	0.05
Zn	14896	59.80	24.71

like arsenic, lead, cadmium and mercury are below the detectable limit and hence strengthened the safety profile.

Table 4: Showing the results of FTIR

Peak values	Functional groups
3840.8	Phenols, Alcohols
2369.2	Carboxylic acid
1653.1	Alkenes
1394.1	Aromatics
1115.9	Aliphatic amines
621.1	Alkynes
538.1	Alkyl halides

The FT-IR analysis of Naga Parpam showed the association of the functional groups and effective peaks were obtained between 4000 cm⁻¹ to 400 cm⁻¹. Existence of functional groups like alcohols, esters, alkanes, aromatic amines was found. These key components may be responsible for the

presence of fertility action of the drug Naga parpam. The XRF analysis of the parpam showed the elements in oxide as well as elemental form. The results are tabulated in the table no.4.as follows.

Table 5: Showing the results of XRFanalysis

Element in oxide form	
Formula	Concentration (%)
Zno	86.99
K ₂ O	8.02
Cl	2.53
SO ₃	1.16
Fe ₂ O ₃	0.54
SiO ₂	0.34
CaO	0.16
Al ₂ O ₃	0.13
Mg	0.09
CuO	0.03

Table 5: Showing the results of XRFanalysis

Elemental form	
Formula	Concentration (%)
Zn	89.51
K	6.66
Cl	2.53
Fe	0.38
Si	0.16
Ca	0.11
Mg	0.09
Al	0.07
S	0.47
Cu	0.02

The elemental as well as oxide form confirmed the absence of heavy metals like arsenic, cadmium, mercury and lead.

4. Conclusion

In the present study it is concluded that the organoleptic characters and physico-chemical parameters such as the total ash value (27.2 %w/w), acid insoluble ash value (11.6%w/w), loss of drying at 105°C (0.29 %w/w) and the pH value (9.6) and can be efficiently used for standardization of polyherbal formulation. The sophisticated analysis of instruments like SEM analysis of the sample showed the presence of nano particles arranged in agglomerates. The quantitative analysis of the sample through EDAX showed the content of minerals like carbon,

oxygen, magnesium, aluminium, silica, sulphur, chloride, potassium, calcium which may be responsible for the pharmacological activity. The FT-IR spectroscopy applied in the mid infra-red region 4000 cm⁻¹ to 400 cm⁻¹ revealed the presence of functional groups like primary aliphatic amines, aromatics, alkynes, esters, alcohols, carboxylic acids which are primarily responsible for the fertility activity. These results could be a better reference for setting limits for the quality assessment of the drug as well as enhancing the therapeutic efficiency of the drug in future.

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