



# Screening Of Physico-Chemical And Heavy Metal Analysis Of Siddha Polyherbal Formulation Nilavagai Chooranam- An Analytical Study

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## Abstract:

Qualitative analysis is the process of ensuring the quality of the drug. Every medicine should be standardized to improve the quality and consistency of the medicine. Currently in Siddha, many medicines like herbal, herbo-mineral formulation and animal products are being standardized. One of the Siddha medicines, Nilavagai chooranam is mentioned for many diseases like, flatulence (Vayutru porumal), vomiting (vaanthi), Hiccup (Vikkal), Peptic ulcer diseases (Gunmam) and etc. In this study qualitative analysis such as macroscopic appearance, physico-chemical analysis including total ash, loss on drying, acid insoluble ash, water and alcohol soluble extractive, pH value and heavy metal analysis were determined. Analysis of Nilavagai chooranam was based on PLIM guidelines. Physico-chemical analysis of chooranam reveals quality and purity of the drug and exhibited the presence of organic bioactive compounds including polyphenols, tannins, saponins and etc. Heavy metal analysis exhibited lead, arsenic, cadmium, mercury is below detectable level. In future, this study will help for further preclinical and clinical evaluation of the drug to treat various health illnesses.

**Key words:** Heavy metal analysis, Nilavagai chooranam, Physico-chemical analysis, Siddha literature, Standardization.

## Introduction:

The traditional system of Siddha medicine are effective, higher safety margin and lower costs. Central council of research in Ayurveda and Siddha has given preliminary guidelines for standardizing these conventional formulations. For the uniformity of batches in production of herbal formulations it is necessary to develop methods for evaluation<sup>1</sup>. The WHO in number of resolutions has emphasized the need to ensure quality control of herbal formulations by using modern techniques and applying suitable standards<sup>2</sup>. Standardization involves the confirmation of the identity, purity, detection of adulterant and analysis of heavy metals through various parameters like macroscopic appearance, physical and chemical tests. Heavy metal analysis was done by AAS (Atomic absorption spectroscopy) method. One of the therapeutic formulations, Nilavagai chooranam is described in classical Siddha literature, Agathiya vaithiya rathina surukkam-360. The chooranam is made up of herbal drugs including *Cassia senna* (Nilavagai), *Piper nigrum* (Milagu), *Trachyspermum ammi* (Omam), *Zingiber officinale* (Sukku), *Embelia ribes* (Vaividangam), *Saccharum officinarum* (Seeni sarkarai)<sup>4</sup>. The preclinical and clinical studies for safety and efficacy will not be possible if authentic drugs are not used<sup>3</sup>. Hence, physico-chemical analysis is the primary step in standardization. Physico-chemical analysis indicates the absence of adulterant and presence of purity, quality of the herbal formulation Nilavagai chooranam. Heavy metal analysis exhibits the level of heavy metals such as lead, arsenic, cadmium, Mercury. This study is aimed to analyze the quality of the drug Nilavagai chooranam to treat various ailments.

### Materials and methods:

The drug selection has been taken from Siddha literature, Agathiya Vaithiya Rathina Surukkam-360, Pg.no: 48. The raw drugs are collected in standard raw drug store, Chennai. Then, the crude drugs are certified by botanists in Gunapadam, Government Siddha medical college, Chennai. After authentication, the drugs were purified based on Sarakkugalin Suthi Seimuraigal. Once purified, the drugs made as chooranam. Nilavagai chooranam was analyzed using macroscopic appearance, physico-chemical parameters such as loss on drying, ash values, water and alcohol soluble extractive, pH value and heavy metal analysis by AAS method was elucidated.

### Method of preparation:

Table no. 1 expresses the ingredients of Nilavagai chooranam. Purified drugs are powdered and sieved in a thin cloth to get fine powder (chooranam). Then, the prepared chooranam further purified under pittaviyal method (steam cooking). Again, it dried and kept in airtight container and labeled as Nilavagai chooranam<sup>4</sup>. Purification of chooranam based on Siddha literature<sup>5</sup>.

Table no. 1. Ingredients of NC

S. No	Ingredients	Quantity of drugs
1.	Nilavagai ( <i>Cassia senna</i> )	35g (1 palam)
2.	Milagu ( <i>Piper nigrum</i> )	35g (1 palam)
3.	Omam ( <i>Trachyspermum ammi</i> )	35g (1 palam)
4.	Sukku ( <i>Zingiber officinale</i> )	35g (1 palam)
5.	Vaividangam ( <i>Embelia ribes</i> )	35g (1 palam)
6.	Seenisarkarai ( <i>Saccharum officinarum</i> )	35g (1 palam)

### Qualitative analysis:

#### A. Macroscopic evaluation:

Appearance, odour, flow property, nature and solubility profile of the Nilavagai chooranam was tested. Fig.1 represents the chooranam colour.

Fig.no 1. Nilavagai chooranam appearance



#### B. Physico-chemical analysis<sup>6,7</sup>:

##### Percentage Loss on Drying

2gm of test drug was accurately weighed in evaporating dish. The sample was dried at 105°C for 5 hours and then weighed.

##### Determination of Total Ash

2gm of test drug was accurately weighed in silica dish and incinerated at the furnace at temperature 400°C until absence of carbon. The sample was cooled and weighed. Percentage of total ash will be calculated with reference to the weight of air-dried drug.

##### Determination of Acid Insoluble Ash

The ash obtained by total ash was boiled with 25 ml of dilute hydrochloric acid for 6 mins. Then the insoluble matter is collected in crucible, washed with hot water and ignited to constant weight. Percentage of acid insoluble ash calculated with reference to the weight of air-dried ash.

### Determination of Alcohol Soluble Extractive

5gm of test sample was macerated with 100 ml of Alcohol in a closed flask for twenty-four hours, shaking frequently during six hours and allowing it to stand for eighteen hours. Filter rapidly, taking precautions against loss of solvent, evaporate 25 ml of the filtrate to dryness in a tared flat bottomed shallow dish, and dry at 105°C, to constant weight and weigh. Calculate the percentage of alcohol-soluble extractive with reference to the air-dried drug.

### Determination of Water-Soluble Extractive

5gm of test sample was macerated with 100 ml of chloroform water in a closed flask for twenty-four hours, shaking frequently during six hours and allowing it to stand and for eighteen hours. Filter rapidly, taking precautions against loss of solvent, evaporate 25 ml of the filtrate to dryness in a tared flat bottomed shallow dish, and dry at 105°C, to constant weight and weigh. Calculate the percentage of water-soluble extractive with reference to the air-dried drug.

### pH determination

Required quantity of test sample was admixed with distilled water and the subjected to screening using pH meter.

### C. Heavy metal analysis:

Atomic Absorption Spectrometry (AAS) is a very common and reliable technique for detecting metals and metalloids in environmental samples. The total heavy metal content of the sample was performed by Atomic Absorption Spectrometry (AAS) Model AA 240 Series. In order to determination the heavy metals such as mercury, arsenic, lead and cadmium concentrations in the test item. Test sample was digested with 1mol/L HCl for determination of arsenic and mercury. Similarly, for the determination of lead and cadmium the sample were digested with 1mol/L of HNO<sub>3</sub>. Standard preparation of chooranam, Arsenic (As) & Mercury (Hg) is 100 ppm sample in 1mol/L HCl and Cadmium (Cd) & Lead (Pb) is 100 ppm sample in 1mol/L HNO<sub>3</sub>.

### Results:

Table no. 2. Summarizes the macroscopic evaluation, Table no. 3. exhibits the solubility profile, Table no. 4. expresses the physico-chemical properties and Table no. 5 indicates the level of heavy metals in Nilavagai chooranam.

Table no. 2. Macroscopic appearance

State	Solid
Nature	Fine powder
Odour	Strong Characteristic
Touch	Soft
Flow Property	Free flowing
Appearance	Brownish

Table no. 3. Solubility profile

S.No	Solvent Used	Solubility / Dispersibility
1.	Chloroform	Insoluble
2.	Ethanol	Soluble
3.	Water	Soluble
4.	Ethyl acetate	Insoluble
5.	DMSO	Soluble

Table no. 4. Physico-chemical parameters

S. No	Parameter	Mean (n=3) SD
1.	Loss on Drying at 105 °C (%)	10.77 ± 0.55
2.	Total Ash (%)	8.367 ± 0.25
3.	Acid insoluble Ash (%)	0.023 ± 0.05
4.	Water soluble Extractive (%)	14.53 ± 0.68
5.	Alcohol Soluble Extractive (%)	8.83 ± 0.472
6.	pH	6.4

Table no. 5. Heavy metal analysis

Metals	Absorbance A Max	Result	Maximum limit range in India
Lead	217.0 nm	3.374	10 ppm
Arsenic	193.7 nm	BDL	3 ppm
Cadmium	228.8 nm	BDL	0.3 ppm
Mercury	253.7 nm	0.108	1 ppm

**Discussion:**

From the above results, the formulation was brownish in colour, having strong characteristic, fine powder in nature, free flowing property. Solubility profile exhibits, the chooranam soluble in water, ethanol and DMSO while insoluble in chloroform and ethyl acetate. Water, ethanol and DMSO solubility expresses the high absorption value to improve bio-availability for achieving pharmacological response<sup>10,11,12</sup>.

Ash value provides inorganic compounds and other impurities present along with the drug. The total ash, acid insoluble ash was found to be  $8.367 \pm 0.25$ ,  $0.023 \pm 0.05$ . This value specifies the authenticity and purity of the sample. The water-soluble extractive and alcohol-soluble extractive value was  $14.53 \pm 0.68$ ,  $8.83 \pm 0.472$ . Hence, the drug was more extracted and indicates the absence of adulterants, correct processing during drying or storage. Loss on drying measured value was  $10.77 \pm 0.55$ . Thus, less moisture content of this drug sensible for higher stability of the chooranam<sup>8</sup>. pH value of the drug (6.4) was slightly acidic in nature.

The Nilavagai chooranam heavy metal analysis showed below detectable level of heavy metals. Thus, the absence of heavy metal contamination such as Arsenic (BDL), Cadmium (BDL), Mercury (0.108 ppm), Lead (3.374 ppm) is compared with the permissible limit of heavy metals in India. Nilavagai chooranam having above quality standards<sup>8</sup>.

**Conclusion:**

The drug Nilavagai chooranam analysis exhibits the purity, quality and authenticity of the ingredients, manufacturing process, storage and free from adulterants. Also, absence of heavy metals demonstrates the safety of the drug. Through this study, further pharmacological research can be done to treat various health illnesses.

**Acknowledgement:**

The author conveys the credits to Staff of Gunapadam department, Dr, K. Kanakavalli, Principal, Govt. Siddha medical college, Chennai, The Tamilnadu Dr.M.G.R. medical university, my colleagues and friends for their guidance and support.

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