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PHYSICOCHEMICAL CHARACTERIZATION AND STANDARDIZATION OF TRADITIONAL *SIDDHA* FORMULATION *MUTHU CHENDURAM*

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ABSTRACT

Background: *Siddha metallo*-mineral formulations such as *Muthu Chenduram* are traditionally valued for their enhanced bioavailability and therapeutic efficacy. However, scientific standardization using modern analytical techniques is essential to validate their safety, composition, and structural transformation.

Objective: To standardize *Muthu Chenduram* through physicochemical evaluation and advanced analytical techniques including ICP–OES, XRD, SEM, and EDAX.

Materials and Methods: The formulation was prepared as per classical *Siddha* procedures described in *Agasthiyar Chenduram 300*, using purified pearl, zinc, mercury, mercury perchloride, sulphur, and milk. Physicochemical parameters were assessed using standard protocols. Elemental analysis was performed using ICP–OES, crystalline phases were identified by XRD, while SEM–EDAX was employed to study morphology and elemental distribution.

Results: The formulation appeared as a red powder with low moisture content (LOD: 2.38%) and high inorganic content (total ash: 23.63%). ICP–OES revealed high levels of mercury (74625.9 mg/kg), zinc, and calcium, with negligible lead and cadmium. XRD confirmed the formation of stable crystalline phases, predominantly mercuric sulphide (HgS), along with ZnS and $CaCO_3$. EDAX analysis corroborated the elemental composition, showing dominant Hg–S complexes. SEM analysis demonstrated nanocrystalline particles (11–18 nm) aggregated into microstructures.

Conclusion: *Muthu Chenduram* is a well-standardized metallo-mineral formulation exhibiting physicochemical stability, nanoscale transformation, and conversion of mercury into a relatively stable sulphide form. The findings support traditional preparation methods and indicate potential for enhanced bioavailability, though further toxicity and pharmacological evaluation are required.

Keywords: *Muthu Chenduram, Siddha medicine, Standardization, XRD, ICP – OES, SEM – EDAX.*

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INTRODUCTION

Siddha pharmaceutics involves unique pharmaceutical processes that convert raw metals and minerals into therapeutically effective and biologically acceptable forms. Among these, *Chenduram* preparations are considered highly potent due to their fine particle size, stability and enhanced bioavailability. *Muthu Chenduram* is a classical *Siddha metallo* – mineral formulation described in the text *Agasthiyar Chenduram300* [1]. The formulation has been indicated for Cancer of Penis, testis & Scrotum, Cervical Cancer, Carbuncle etc., In recent years, the need for scientific validation and standardization of traditional medicines has gained importance. Modern analytical techniques such as Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP – OES), X-ray Diffraction (XRD), Energy Dispersive X-ray Analysis (EDAX) and Scanning Electron Microscopy (SEM) provide valuable insights into elemental composition, concentration, surface characteristics and structural transformations. The present study aims to standardize *Muthu Chenduram* by integrating classical *Siddha* concepts with modern analytical validation.

MATERIALS AND METHODS

Authentication and Purification

All raw drugs were authenticated by the experts of *Gunapadam* (*Siddha* Pharmacology). The ingredients were purified as per the *Siddha* text *Sarakku SuddhiSei Muraigal* [2].

Preparation of *Muthu Chenduram*

Purified pearl, zinc, hydrargyrum and hydrargyrumperchloride (35 gram each) were triturated with sufficient quantity of milk using a stone mortar and pestle for one day. The mixture was pelletized and dried under sunlight. The dried pellets were further triturated with 70 grams of sulphur and again made into pellets. These pellets were placed in an earthen container, sealed with mud plaster and subjected to incineration with 10 cow dung cakes (*Kozhi Pudam*). After self-cooling, the pellets were removed, powdered and preserved in an airtight container.

Physicochemical Analysis

All the physicochemical parameters were carried out as per the standard test procedures LOHAR, Protocol for Testing of Ayurvedic, Siddha and Unani Medicines, 2008 [3].

ICP – OES Analysis

The analysis done as per the guidelines mentioned in Official Methods of Analysis of AOAC International, 2016 [4].

Powder XRD Analysis

Powder diffraction data were collected by Aeris PAN alytical diffract meter (Netherlands) with Ni – filtered copper radiation in Bragg – Brentano geometry. Fine powder of the sample taken in a thin layer on a silicon zero background holder. The sample was recorded for the angle 2θ in the range of 10 – 90 degrees at a scanning rate of 4 degrees/sec with CuKα (λ 1.5418 Å).

SEM - EDAX

The formulation was finely powdered and mounted on aluminium stubs using conductive carbon tape, followed by gold sputter coating under vacuum to enhance conductivity. Scanning Electron Microscopy (SEM) was performed using a Zeiss Gemini 560 Field Emission SEM at an accelerating voltage of 10 kV to evaluate particle size, morphology, and surface characteristics at various magnifications. Energy Dispersive X-ray Analysis (EDAX), coupled with SEM, was used to determine elemental composition. Spectra were obtained from multiple regions, and elements were identified along with their weight and atomic percentages. Elemental mapping was carried out to assess the distribution of elements.

RESULTS

Organoleptic Characters

Colour: Brick Red

Texture: Powder

Table 01: Physicochemical Parameters

S.No	Parameter	Result
1.	Loss On Drying (LOD)	2.38%
2.	Total Ash	23.63%
3.	Water Soluble Ash	20.57%
4.	Acid Insoluble Ash	4.93%
5.	Water Soluble Extractives	2.53%
6.	Alcohol Soluble Extractives	5.27%
7.	pH	6.26

ICP – OES

Table 02: Physicochemical parameters of Muthu Chenduram

S.No	Element	Result (mg/Kg)
1.	Arsenic (As)	2.99
2.	Cadmium (Cd)	BLQ
3.	Mercury (Hg)	74625.9
4.	Lead (Pb)	BLQ
5.	Calcium (Ca)	27643.88
6.	Zinc (Zn)	59424.56

XRD

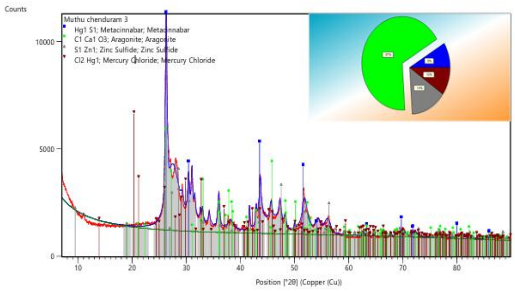

Figure 01: Powder X-ray diffraction (XRD) pattern of Muthu Chenduram showing major crystalline phases.

EDAX

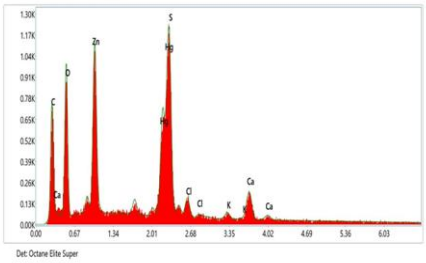

Figure 02: EDAX spectrum depicting the elemental composition of Muthu Chenduram.

SEM

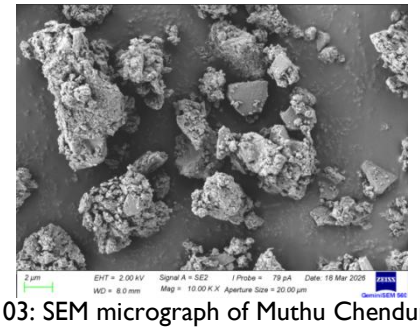

Figure 03: SEM micrograph of Muthu Chenduram at lower magnification showing agglomerated particles.

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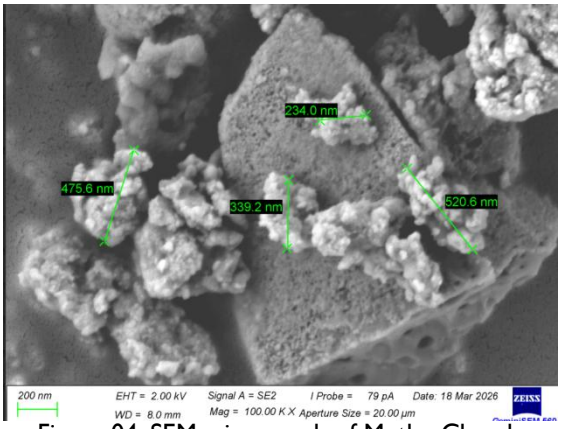

Figure 04: SEM micrograph of Muthu Chenduram showing particle size distribution at intermediate magnification.

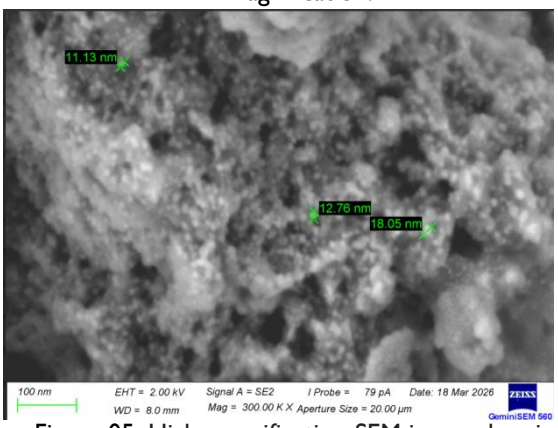

Figure 05: High-magnification SEM image showing nanocrystalline particles of Muthu Chenduram.

DISCUSSION

Organoleptic and Physicochemical Evaluation

The organoleptic and physicochemical parameters indicate that the formulation is a well-prepared metallo-mineral drug. The brick red coloration and fine powdery nature suggest efficient calcination and particle size reduction, which are critical for enhancing bioavailability. The low loss on drying (2.38%) reflects minimal moisture content, ensuring stability and reduced risk of microbial contamination. The total ash value (23.63%) confirms the predominance of inorganic constituents. The high water-soluble ash (20.57%) indicates that a significant fraction of inorganic components is readily soluble, suggesting improved absorption potential. The acid-insoluble ash (4.93%) remains within acceptable limits, indicating minimal siliceous contamination and effective purification. Extractive values reveal a lower water-soluble fraction (2.53%) and a comparatively higher alcohol-soluble fraction (5.27%), suggesting the presence of semi-polarconstituents. The near-neutral pH (6.26) indicates suitability for internal administration without significant gastric irritation.

ICP–OES Analysis

Arsenic (2.99 mg/kg) is present in low concentration, slightly greater than permissible limits, while cadmium and lead were below the limit of quantification, indicating negligible contamination and effective purification.Mercury content (74625.9 mg/kg) is markedly high,where mercury is intentionally incorporated and processed. Elevated levels of calcium (27643.88 mg/kg) and zinc (59424.56 mg/kg) suggest potential roles in structural integrity and pharmacological activity, particularly in enzymatic and immunomodulatory functions.

XRD Analysis

The diffraction peaks correspond predominantly to metacinnabar (HgS), indicating mercury stabilization in sulphide form, which is considered relatively less toxic. Peaks corresponding to zinc sulphide (ZnS) and aragonite ($CaCO_3$) further confirm the presence of stable mineral phases.Minor peaks of mercury chloride ($HgCl_2$) suggest trace intermediate or residual compounds. The sharp and well-defined peaks indicate high crystallinity and effective calcination, while the absence of amorphous patterns suggests minimal unreacted material. Overall, XRD findings confirm successful conversion of raw materials into stable crystalline phases, supporting reduced toxicity and therapeutic potential.

EDAX Analysis

EDAX analysis validates the elemental composition and corroborates ICP–OES findings. Prominent peaks of mercury (Hg) and sulphur (S) indicate the formation of mercuric sulphide (HgS). Significant zinc (Zn) and calcium (Ca) peaks suggest their involvement in therapeutic activity and structural stabilization.Minor peaks of carbon and oxygen indicate residual organic components, while chlorine and potassium represent trace inorganic salts. The absence of detectable lead and cadmium supports the purity of the formulation. Overall, EDAX confirms a well-processed metallo-mineral matrix dominated by Hg–S complexes with supportive elements.

SEM Analysis

SEM analysis reveals the surface morphology and particle size distribution at varying magnifications. At lower magnification (100 KX), irregular agglomerated particles ranging from 234 nm to 520.6 nm are observed, indicating clustering during calcination. These aggregates exhibit rough and porous morphology, enhancing surface area and potential bio interaction. At higher magnification (300 KX), nanosized particles (11.13–18.05 nm) are evident, forming a hierarchical structure where primary nanoparticles aggregate into secondary clusters. This nanocrystalline nature reflects effective trituration and incineration processes. Overall, the formulation exhibits a dual-scale structure consisting of nanoparticles embedded within micro-sized aggregates, which may enhance dissolution, absorption, and therapeutic efficacy.

CONCLUSION

The present study demonstrates that the prepared *Muthu Chenduram* is a well-processed metallo-mineral formulation with desirable physicochemical and

structural characteristics. The organoleptic and physicochemical parameters confirm its purity, stability, and suitability for internal administration. Elemental analysis by ICP–OES indicates a mineral-rich composition with negligible levels of toxic heavy metals such as lead and cadmium, while the presence of mercury in high concentration aligns with traditional formulations and warrants evaluation of its chemical form.XRD analysis confirms the successful transformation of mercury predominantly into a stable sulphide (HgS) phase, supported by EDAX findings, indicating proper calcination and reduced toxicity. SEM analysis further reveals a nanocrystalline structure with aggregated particles, suggesting enhanced surface area and potential bioavailability. Overall, the findings validate the effectiveness of the traditional preparation method in achieving physicochemical stability, nanoscale transformation, and mineral phase stabilization. However, further studies on mercury speciation, toxicity profiling, and pharmacological evaluation are essential to establish the safety and therapeutic efficacy of the formulation.

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CONFLICT OF INTEREST

Not declared.

INFORMED CONSENT

Not applicable

ETHICAL STATEMENT

Not declared

AUTHOR CONTRIBUTION

The research article written and self funded by Karthikaa T under the guidance of Lakshmanaraj C.

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