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Research Article

PHYSICOCHEMICAL CHARACTERIZATION OF *SOODHA GANDHI DHARA CHENDURAM* BY X-RAY POWDER DIFFRACTION: CONFIRMATION OF CINNABAR AS THE PREDOMINANT CRYSTALLINE PHASE

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ABSTRACT

Soodha Gandhi Dhara Chenduram is a classical *Siddha* mercurial formulation (*Chenduram*) prepared using purified mercury (*Soodham*), Sulphur (*Gandhagam*), and arsenic trisulphide (*Aridhaaram*) in a ratio of 3:1: ½ through the traditional *Kajjali* preparation followed by subsequent incineration. The present study aimed to characterize the crystalline phases formed during the *Siddha* preparation process using X-ray Powder Diffraction (XRD), thereby providing physicochemical evidence for the transformation of the starting materials. XRD analysis revealed cinnabar (HgS, α-phase) as the predominant crystalline phase, with a score of 80 and a scale factor of 0.791. Dipotassium Sulfate Alpha (K_2SO_4) was identified as a minor crystalline phase, with a score of 22 and a scale factor of 0.121. The diffraction pattern exhibited a dominant peak at 2θ=31.14° with a d-spacing of 2.87 Å and relative intensity of 100%, along with a major peak at 2θ=26.44° with a d-spacing of 3.37 Å and relative intensity of 86.46%. These reflections are consistent with the characteristic diffraction pattern of trigonal Cinnabar. The predominance of HgS demonstrates the conversion of mercury and sulphur into a crystalline mercury sulphide phase during the traditional processing steps. The findings support the occurrence of substantial physicochemical transformation during *Kajjali* formation and subsequent incineration, resulting in a predominantly crystalline HgS-containing product. The identification of defined crystalline phases provides useful information for physicochemical characterization, quality assessment, and standardization of *Soodha Gandhi Dhara Chenduram*. The study also contributes to the scientific understanding of the traditional Siddha pharmaceutical process and provides a basis for further investigations into its physicochemical properties and therapeutic relevance.

INTRODUCTION

Chenduram is a distinct class of red-coloured, calcined mineral formulations in *Siddha* medicine, prepared by subjecting metals, minerals or their combinations to specific incineration procedures. Unlike *Parpam, Chenduram* formulations typically involve mercurial or sulphide-based starting materials and are characterised by a brick-red to vermilion colour.

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The pharmacological profile of *Chenduram* preparations is considered potent in the *Siddha* therapeutic tradition with carefully prescribed doses and vehicles.

Soodha Gandhi Dhara Chenduram is a well-documented formulation in classical *Siddha* text *Theraiyar Vaithiyam 1001* prepared from three ingredients: *Soodham* (purified mercury, Hg), *Gandhagam* (sulphur, S), and *Aridharam* (arsenic trisulphide, As_2S_3), taken in the ratio of 3:1: ½ respectively.[1] The preparation follows a two-stage process. First, the formation of *Kajjali* (a black mercury–sulphur amalgam) through trituration, followed by addition of *Aridharam* and incineration in a sealed earthen crucible. The formulation is traditionally indicated in fever *(Suram),* skin disorders *(Thol Noi),* rheumatic conditions (*Vadha soolai*)

administered with appropriate vehicles (*Anubanam*) such as rice gruel, milk, honey water or plain water.

Despite its documented clinical use, there is a paucity of modern analytical data characterising the structural and phase composition of *Soodha Gandhi Dhara Chenduram*. X-ray Powder Diffraction (XRD) is the most reliable technique for phase identification in such complex mineral formulations.[2] The present study aims to characterise the crystalline phases of *Soodha Gandhi Dhara Chenduram* using XRD analysis, thereby providing a scientific basis for understanding its physicochemical nature.

MATERIALS AND METHODS

Raw Materials

The three ingredients used in the preparation were: (i) Mercury (*Soodham*, Hg) - 3 parts (60 g); (ii) Sulphur (*Gandhagam*, S) - 1 part (20 g); (iii) Arsenic trisulphide (*Aridharam*, As_2S_3) - ½ part (10 g), giving a total ratio of 3:1: ½ (6:2:1 by weight proportion). [3] All raw materials were procured from an esteemed *Siddha* raw drug store. It was then authenticated by the experts of *Siddha* Pharmacology.

Preparation of *Soodha Gandhi Dhara Chenduram*

The preparation was carried out strictly in accordance with the classical *Siddha* textual procedure. In the first stage, sulphur (*Gandhagam*) was melted and mercury (*Soodham*) was added into the molten sulphur. The mixture was triturated continuously on a granite stone mortar and pestle until a homogeneous, jet-black powder (*Kajjali*) was obtained, indicating complete reaction between mercury and sulphur. In the second stage, arsenic trisulphide (*Aridharam*) was added to the *Kajjali* and the combined mass was triturated thoroughly. The resulting mixture was formed into pellets, placed in an earthen crucible, and sealed with clay. The sealed crucible was subjected to bio incineration using a controlled fire. The resulting red powder (*Chenduram*) was collected, weighed, and stored in an airtight container for analysis.

XRD Analysis

XRD analysis of *Soodha Gandhi Dhara Chenduram* was performed on 03 June 2026 at Siddha Central Research Institute, Arumbakkam, Chennai. It was analysed using a panalytical X-ray diffractometer (Diffractometer Type: 348709; Goniometer Radius: 145.00 mm) with Cu Kα radiation (K-Alpha1: 1.54060 Å, K-Alpha2: 1.54443 Å), generator settings of 40 kV and 8 mA, scan range 7.01°–89.97° (2θ), step size 0.022° (2θ), scan step time 90.27s, divergence slit 0.4584°, and specimen length 10mm.[4] The measurement was conducted at 25°C under continuous scanning mode with PSD scanning. Phase identification was performed using High Score Plus software against the ICDD reference database.

RESULTS

XRD Diffractogram

The XRD diffractogram of *Soodha Gandhi Dhara Chenduram* showed a complex pattern with 35 diffraction peaks across the 2θ range of 7°–90° (Figure 1), indicative of a multi-phase, well-crystalline product.[5] The two most intense peaks were recorded at 2θ= 31.14° (relative intensity 100%, d-spacing 2.87 Å) and 2θ=26.44° (relative intensity 86.46%, d-spacing 3.37 Å). Other notable peaks appeared at 2θ=28.11° (26.17%), 43.55° (19.79%), 45.72° (17.44%), 54.54° (13.71%), 52.66° (13.47%), and 51.69° (13.09%). The notably broad FWHM values for many peaks (0.17°–0.48°) in the higher 2θ region suggest moderate to smaller crystallite size in those phases.

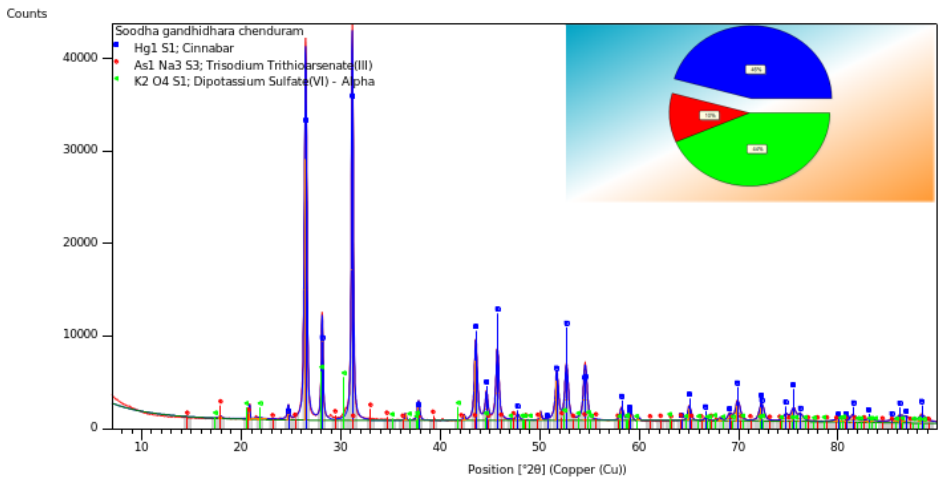


Figure 1: XRD diffractogram of *Soodha Gandhi Dhara Chenduram* showing phase identification with Cinnabar (Hg1 S1), Trisodium Trithioarsenate (III) (As1 Na3 S3), and Dipotassium Sulfate–Alpha (K2 O4 S1) reference patterns. The inserted pie chart shows the relative phase distribution

Phase Identification

Phase matching against the ICDD reference database (table 1) identified the following crystalline phases:

1. Cinnabar (α-HgS) - Ref. Code: 98-005-6475; Score: 80; Scale Factor: 0.791; Chemical Formula: HgS. This was the dominant phase, contributing the majority of peaks including the two most intense reflections at 2θ = 31.14° and 26.44°.
2. Dipotassium Sulfate–Alpha (K_2SO_4)- Ref. Code: 98-003-1534; Score: 22; Scale Factor: 0.121; Chemical Formula: K_2SO_4. This phase was identified as a minor constituent. The moderate match score (22) warrants cautious interpretation; the assignment is considered tentative and should be confirmed by complementary techniques such as FTIR or EDS.
3. Trisodium Trithioarsenate (III) (Na_3AsS_3)- Ref. Code: 98-000-1036; Score: 13; Scale Factor: 0.032; Chemical Formula: Na_3AsS_3. This phase showed a very low match score (13) and must be regarded as a tentative identification only. No strong mechanistic basis exists for sodium incorporation from the current preparation materials; further analysis is required to confirm or exclude this phase.

Table 1: Selected XRD Peaks of Soodha Gandhi Dhara Chenduram (peaks with Rel. Int. > 3%)

2θ (°)	Height (cm)	FWHM (°2θ)	d-spacing (Å)	Rel. Int. (%)	Assigned Phase
20.81	1162.11	0.1213	4.2658	3.56	Cinnabar
24.70	1123.56	0.1787	3.6017	3.44	Cinnabar
26.44	28217.06	0.2633	3.3680	86.46	Cinnabar
28.11	8539.80	0.1700	3.1719	26.17	Cinnabar
31.14	32637.22	0.1709	2.8702	100.00	Cinnabar (dominant)
37.79	1623.12	0.2329	2.3787	4.97	Dipotassium Sulfate
43.55	6457.61	0.2830	2.0764	19.79	Cinnabar
44.62	2204.76	0.2608	2.0291	6.76	Cinnabar
45.72	5693.39	0.3016	1.9830	17.44	Cinnabar
51.69	4270.63	0.2532	1.7670	13.09	Cinnabar
52.66	4397.19	0.3377	1.7367	13.47	Cinnabar
54.54	4475.28	0.3644	1.6813	13.71	Cinnabar
69.87	1610.53	0.4811	1.3452	4.93	Dipotassium Sulfate
72.26	1421.45	0.4859	1.3065	4.36	Dipotassium Sulfate

Note: Full peak list (35 peaks) is provided in the raw XRD data. Table 1 presents peaks with relative intensity > 3% for clarity.

DISCUSSION

The XRD analysis of *Soodha Gandhi Dhara Chenduram* confirmed cinnabar (α-HgS) as the primary crystalline product, with a high match score of 80 and scale factor of 0.791. This finding is chemically consistent with the preparation methodology. The first and most critical step in the preparation is the formation of *Kajjali* the black amalgam of mercury and sulphur formed by prolonged trituration. During this stage, mercury and sulphur undergo mechanochemical reaction to form black metacinnabar (β-HgS). Upon subsequent incineration at elevated temperatures, β-HgS undergoes phase transformation to the thermodynamically stable red α-HgS (cinnabar), which is reflected by the characteristic brick-red colour of the final *Chenduram* and confirmed by the dominant cinnabar peaks in the XRD pattern.

The two dominant peaks at 2θ=31.14° (d=2.87 Å) and 26.44° (d=3.37 Å) correspond to the (110) and (101) planes of trigonal Cinnabar (space group $P3_121$), consistent with ICDD reference 98-005-6475. The broad cluster of peaks between 2θ=43°–55° is characteristic of the denser reflections in the Cinnabar structure and further supports this assignment.

The presence of Dipotassium Sulfate–Alpha (K_2SO_4, score 22) as a minor phase is noteworthy. Potassium may originate from the clay soil *(Sivappu Mann)* used during sealing of crucible, as many red clay soils contain potassium-bearing minerals. However, the moderate match score of 22 means this assignment should be treated as tentative. Further confirmation by FTIR spectroscopy or Energy Dispersive X-ray Spectroscopy (EDS) is recommended.

The third matched phase, Trisodium Trithioarsenate (Na_3AsS_3, score 13), must be regarded with caution. The very low match score of 13 is below the threshold for confident phase identification. While

arsenic is present in the formulation as *Aridharam* (As_2S_3), there is no clear source of sodium in the preparation ingredients. This match may represent overlapping peaks from the Cinnabar or sulphate phase rather than a genuinely distinct compound. Additional characterisation particularly EDS elemental mapping is needed before any firm conclusion can be drawn about arsenic speciation in the final product.

Importantly, the complete absence of elemental mercury (Hg) peaks in the diffractogram confirms that free mercury has been fully converted during the preparation process, a critical safety and quality parameter for any mercurial *Siddha* formulation.[6] Similarly, the absence of unreacted sulphur peaks indicates complete incorporation of sulphur into the HgS matrix. The Cinnabar based composition of *Soodha Gandhi Dhara Chenduram* is consistent with its traditional therapeutic profile, as cinnabar has been studied for its use in fever management and skin disorders in traditional medicine systems across cultures. [7]

This is a baseline preliminary investigation using XRD. This study alone is inconclusive about the elements present in *Soodha Gandhi Dhara Chenduram*. However, it must be noted that cinnabar contains mercury. Therefore, safety, toxicity and bioavailability studies are essential before any clinical translation.

CONCLUSION

XRD characterisation of *Soodha Gandhi Dhara Chenduram* confirmed cinnabar (α-HgS) as the dominant crystalline phase, validating the *Kajjali* formation and incineration steps of the classical *Siddha* preparation. Dipotassium sulfate was identified as a tentative minor phase, possibly originating from clay used during processing. The trisodium trithioarsenate match was of low confidence and requires further verification. The absence of free mercury and unreacted sulphur in the final product demonstrates the completeness of the preparation process. The phase composition is consistent with the traditional therapeutic applications of the formulation. Further studies including FTIR, SEM-EDS, particle size analysis, and in vitro safety profiling are recommended to comprehensively characterise this formulation and support its evidence-based documentation.

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