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STANDARDISATION OF AYA CHENDURAM, A NANOSIZED HERBO-MINERAL SIDDHA FORMULATION USING MODERN TECHNIQUES

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ABSTRACT

Standardization of Siddha preparations is an important task in establishing the safety and efficacy of the drug. The World Health Organization (WHO) guidelines on evaluating the physico-chemical properties and other parameters for the identification of AYUSH formulations which will offer a great value in global market. Characterization of Siddha formulation renders wide range of information in predicting the nature and structure of phytoconstituents which renders the actual therapeutic efficacy of the formulation. The main aim of the present study is to standardize Aya Chenduram, a nano sized formulation, and also to characterize the same by using sophisticated techniques like Fourier transform infrared spectroscopy (FTIR), Raman Spectroscopy, X – ray diffraction (XRD) and Scanning Electron Microscope (SEM) with EDAX. The X-ray diffraction studies identified that the major crystalline material present in the formulation was alpha-iron oxide. FTIR and Raman spectral data are also supported the presence of alpha iron oxide in Aya chenduram. Further, the results obtained from the SEM analysis shows that most of the particles present in Aya chenduram are in nano and near nano size ranging from 79 nm to 83 nm. In conclusion, data retrieved from the current characterization study of Aya chenduram confirms the size, shape and nature of nano-components and hence this could be used as standards for evaluating the quality of Aya chenduram in future.

KEYWORD: Phytoconstituents, physico-chemical.

INTRODUCTION

Siddha system of medicine is always peculiar due to the use of metals and minerals in their preparations. The metal/ mineral drugs are treated with herbs which are given as bhasmas and chendurams. Bhasmas are fine ash obtained through incineration. Chendurams are prepared by the process of sublimation and they are very much potent medicines. Through process of purification and processing, the metals are becoming biocompatible. In the herbo-mineral preparations, the metals are transforming into very potent drugs.[1,2]

India has very long, safe and continuous usage of many herbo-mineral drugs. But nowadays, there is questions arised on the safety and efficacy behind these herbo-mineral preparations. One of the major bottlenecks in the wider acceptance of these drugs from developing countries is the inadequacy or lack of standardization. Standardization ensures the quality of medicines and gives authenticity to the medicines prepared by the manufacturers, satisfaction of the prescribing physicians and relief to the consumers.

In present investigation, validation of a Siddha herbo-mineral preparation Aya chenduram was done by analyzing the physico-chemical properties. Though, this medicine has been traditionally used, till there is no scientific validation reported for this formulation.

Modern techniques such as XRD, SEM with EDAX, FTIR and Raman spectral studies were used to generate physico-chemical fingerprint for Aya chenduram, prepared as per the Siddha classical text by the process of calcination. Aya chenduram is used in the treatment of various disorders such as insufficiency of seminal secretions (Oligospermia), Nocturnal emission (Spermatorrhoea) and premature ejaculation. Aya chenduram is also given in anaemia, jaundice, oedema, ascites, liver diseases and rheumatism. Dose

recommended is 100-200 mg twice or thrice a day with honey or ghee.[3]

In present study, the evidences were established to ensure the identity of Aya chenduram. The FT-IR spectroscopy showed that the major stretching vibrations of different functional groups in organic compounds in the spectra have very low intensity. During the heating process involved in the preparation of the formulation, the organic groups might have changed into gaseous oxidized compounds and might have escaped. The X-ray diffraction studies identified that the major crystalline material present in the formulation was alpha-iron oxide. The SEM-EDAX study gave important clues regarding the morphology and composition of the sample. Some particles in nano range were also identified.

Nanotechnology provides the tools and technology platform for the investigation of bio-assembled components. Nanobiotechnology is defined as a field that applies the nanoscale principle and techniques to understand bio systems and which uses biological principles and materials to create new devices and systems integrated from the nanoscale.[4]

MATERIALS AND METHODS

1. Aya chendooram

The Siddha classical medicine Aya chendooram was procured from IMCOPS (The Indian Medical Practitioner's Co-operative Pharmacy & Stores Ltd.). The drug was prepared by the methods of Agasthiyar Paripoornam – 400 & Siddha Vaidhya Thirattu. The ingredients are (1) Purified Iron filings, (2) Vinegar and (3) Odina (*Odina woodier* Roxb.) bark juice. Odina bark juice was extracted by adding vinegar and pounding. Take strained juice and put iron filings in it and keep in the sun for 3 days, stirring the mixture as much as possible. Then grind with more of the juice, make cakes, dry and calcine eight times repeating grinding in the juice every time. Then calcine three more times using odina bark juice extracted without adding vinegar. Collect the final product and finely powder and store. The chendooram should be of a dark tan colour.[3]

2. Organoleptic and Physical properties

The organoleptic characters such as colour, touch, taste and odour were noted. To assess the physical properties, the chendooram was subjected to the physical tests like ability of chenduram to float on water to ensure lightness; particle must be inserted in furrows of finger of human hand to ensure particle size and freeness of particles from adhesives to each other.[5,6,7]

3. Physico-chemical parameters

Aya chendooram was subjected for the determination of physic-chemical parameters such as total ash, acid-insoluble ash and water soluble ash using standard methods.[8]

Inorganic qualitative analysis

Inorganic qualitative analysis was carried out by standard laboratory methods.

4. FT-IR (Fourier Transform-Infra Red) spectral analysis

FT-IR spectral analysis was carried out by KBr method[9] using Bruker Optik GmbH FT-IR spectrometer, Germany Model No.: TENSOR 27 with Middle-infrared light (MIR) source. The FTIR spectra of the sample were recorded between 400 cm^{-1} and 4000 cm^{-1}.

5. Raman spectral analysis

The analysis was carried out using Renishew Invia Reflex Spectrometer, focal length 250 mm and Raman Spectrum at 50 cm^{-1} to 4000 cm^{-1}.

6. XRD analysis

The XRD studies were carried out by Bruker D8-Advance X-ray diffractometer (Cu-Kα radiation; λ= 1.5405 Å).[10]

7. SEM (Scanning Electron Microscopy) and EDAX (Energy Dispersive analysis of X-ray) analysis

The elemental composition of the sample was determined Carl Zeiss, Germany Model: SUPRA 55VP, Gemini Column FESEM with air lock system with Energy Dispersive X-ray Analysis (Edx) Oxford Instruments X-MAX (20mm^2). A representative portion of each sample was sprinkled onto a double side carbon tape and mounted on Aluminium stubs, in order to get a higher quality secondary electron image for SEM and EDAX examination. The SEM analysis gives the surface morphology, grain size, particle size, distributions, material homogeneity and inner metallic distributions.

RESULTS AND DISCUSSION

Aya chendooram is a fine powder of dark tan colour. The Loss on drying was found to be 0.518%, Total ash value 98.27%, Water soluble ash 96.87% and Acid insoluble ash 1.40%.

The qualitative analysis showed the presence of Chloride, Sulphate, Sodium, and Iron.

The FT-IR spectrum is given in Fig. 1. The absorption peak in the range of 3900 – 3600 cm^{-1} observed corresponds to the stretching vibration intermolecular hydrogen bond (O – H) existing between the adsorbed water molecules and indicates the lower amount of hydroxyl group. The two peaks at 563 – 472 cm^{-1} appear in IR spectrum shows the presence of Fe - O vibration mode of hematite. No peak at 2900 cm^{-1} indicating the C – H stretching band which means all organic compounds are removed from the samples after calcinations at 700ºC. The characteristic peak at 563 cm^{-1} indicating the formation of stretching mode of α – Fe_2O_3 which specifies the occurrence of α – Fe_2O_3 in calcined compounds.

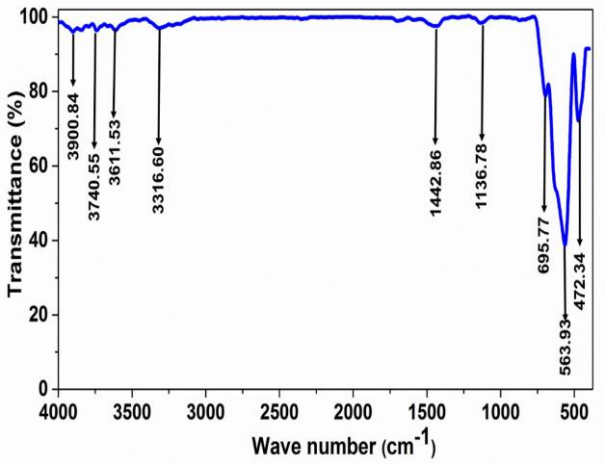

Figure 1: FTIR spectrum of Aya chenduram.

Raman spectroscopy is a spectroscopic technique used to observe vibrational, rotational, and other low-frequency modes in a system.[11] Raman spectroscopy is commonly used in chemistry to provide a structural fingerprint by which molecules can be identified. The Raman spectrum of the Aya chenduram is presented in Fig. 2. In the range of 200–1500 cm^{-1}, many peaks could be observed, which were located at 225, 290, 405, 495, 611, 658 and 1322 cm^{-1}, respectively. All these peaks correspond to α-Fe_2O_3 phase.[12-16] The Raman peaks appearing at 225 and 495 cm^{-1} were assigned to A_1g mode, and those at 290, 405, and 611 cm^{-1} were assigned to E_g modes. The peak located at 658 cm^{-1} is attributed to disorder effects and/or the presence of Fe_2O_3 nanocrystals, while the peak observed at 1322 cm^{-1} has been assigned to hematite two-magnon scattering.[12-14]

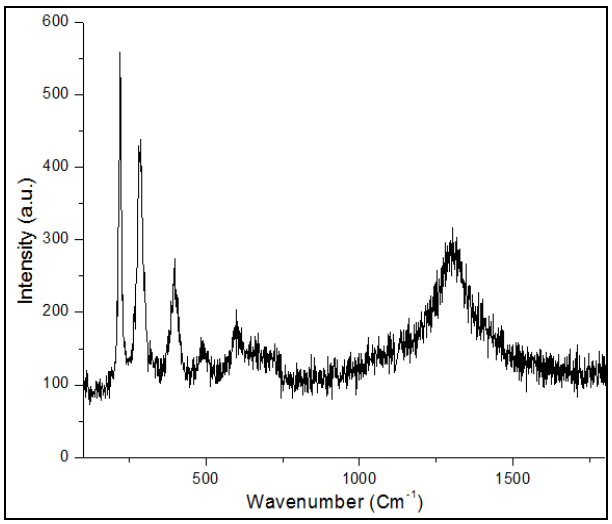

Figure 2: Raman spectrum of Aya chendooram.

X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The X-ray diffraction pattern of the Siddha medicine Aya chenduram (Figure 3) existed with major Fe_2O_3 pattern and its major peaks exactly matched with the JCPDS (Joint Committee on Powder Diffraction Standards) data card number (JCPDS-33-0664). It can be seen from Fig. 3 that eight characteristic peaks observed for Aya Chenduram (2θ= 24.14º, 33.15, 35.61º, 40.85º, 49.48º, 54.16º, 62.82º and 63.99º) can be attributed to the (012, 104, 110, 113, 024, 116, 214 and 300) crystalline structures which corresponds to pure α – Fe_2O_3.

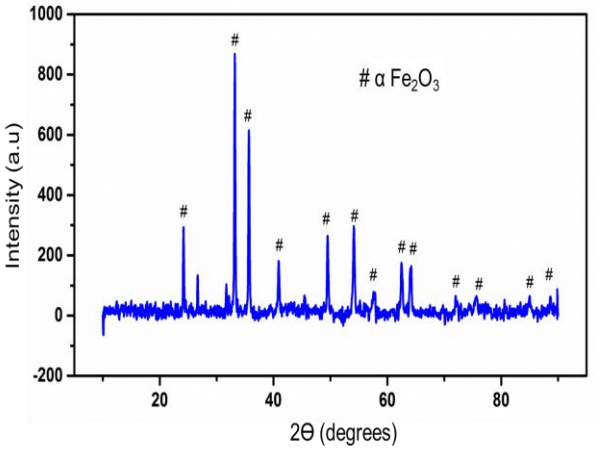

Figure 3: XRD pattern of Aya chendooram.

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with particles in the sample, producing various signals that contain information about the sample's surface topography and composition. The electron beam is scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer[17]. The SEM images clearly indicated the presence of aggregated particle formation with alpha-iron oxide as a major phase (Figure 4). The SEM images showed difference in size and grain of nanoparticles. Nanoparticles are particles between 1 and 100 nanometres (nm) in size. The results obtained from the SEM analysis shows that most of the particles present in Aya chenduram are in nano and near nano size ranges from 79 nm to 83 nm. The nanoparticles in the formulation may be due to the upgradation of crystalline phase in the powder during the calcination. The materials showed good crystalline in nature with particle size of below 100 nm, which was also predicted and confirmed by SEM analysis. It is widely believed that formulation containing nano particle will effectively bind with the bacterial membrane and will aid in penetrating the cellular component of the organism thereby preventing its replication.

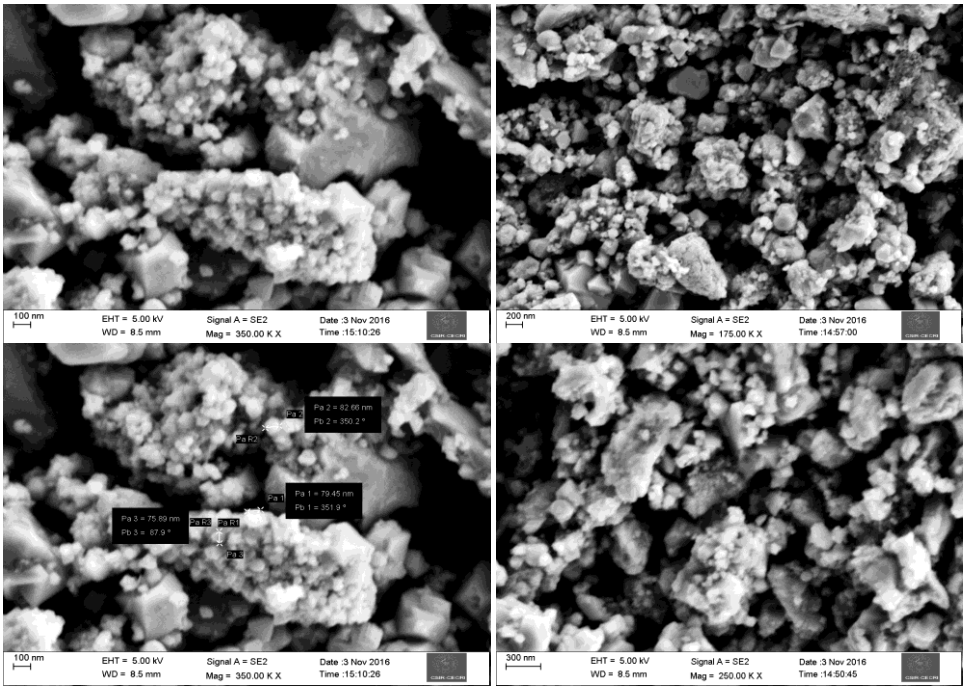

Figure 4: SEM pattern of Aya chendooram.

Elemental composition was determined by Energy dispersive X-ray spectroscopy (EDAX). EDAX is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction between some source of X-ray excitation and the sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum.[18]

Table 1: Elemental composition of Aya chendooram using EDAX.

Sl. No.	Element	Weight%	Atomic%
1.	C (Carbon)	6.25	13.83
2.	O (Oxygen)	34.41	57.19
3.	Na (Sodium)	0.59	0.68
4.	Si (Silicon)	0.25	0.24
5.	S (Sulphur)	0.25	0.21
6.	Cl (Chloride)	0.41	0.31
7.	Fe (Iron)	57.84	27.54

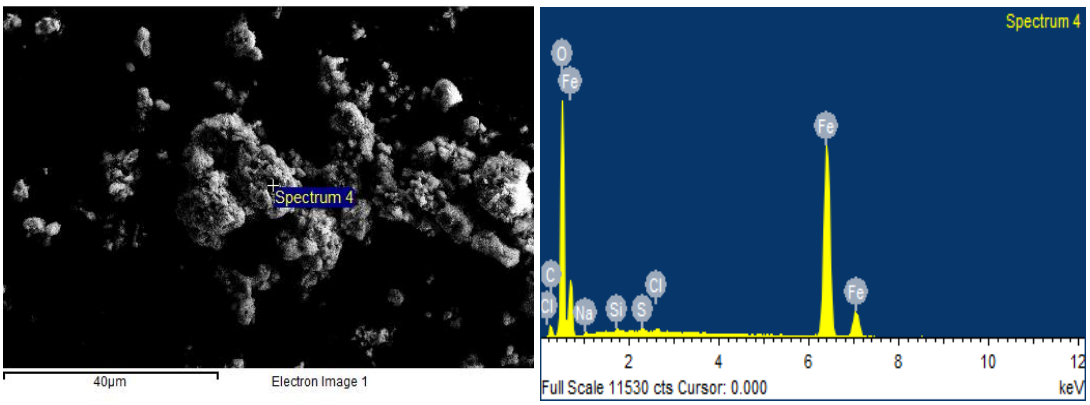

Figure 5a: Electron image of Aya chendooram using SEM; Figure 5b: Elemental composition of Aya chendooram using EDAX.

The results of EDAX analysis are given in Table 1. EDAX provides a good estimate of the concentration of the main elements in the sample. EDAX data shows the exact amount of respective element present in the sample in terms of w/w % (Figure 4). Iron is the major component of the herbal formulation compared to all other minor elements such as carbon and siliceous materials, which existed in minor quantity and therefore, it will not create any major change in overall activity (Figures 5a and 5b).

CONCLUSION

The present study reveals that the heavy/ toxic metals As, Pb, Cd and Hg were found to be absent. This ensured the safety of this formulation. XRD studies confirmed the alpha iron oxide phase existed in the formulation. SEM analysis shows the aggregated particle morphology

for the formulation. The above investigation on Aya chenduram using modern techniques establishes the fingerprint for standardization of the herbal formulation.

In this study Aya chenduram, a mineral based Siddha classical medicine is characterized scientifically for elemental composition, structural and textural properties, morphology and crystalline structure by different characterization techniques. The EDAX analysis shows the presence of Carbon, Oxygen, Sodium, Silicon, Sulphur, Chloride and Iron. FT-IR spectroscopy shows that the major stretching vibrations of different functional groups in organic compounds in the spectra have very low intensity. During the heating process involved in the preparation of the formulation, the organic groups might have changed into gaseous oxidized compounds and might have escaped. The XRD shows that the major crystalline material present in the chenduram is α-Fe_2O_3.

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