INTERNATIONAL
AYURVEDIC
MEDICAL JOURNAL

IAMJ

Research Article ISSN: 2320 5091 Impact Factor: 4.018

CHARACTERISATION OF AYAKANTHA CHENDURAM, A NANOSIZED HERBO-MINERAL FORMULATION USING MODERN TECHNIQUES

Gayathri Devi V[1], Anitha John[1], Natarajan M[2], Neethu Kannan B[3] and Kanagarajan A[4]

[1]Research Officer (Chemistry), [2]Research Assistant (Chemistry), [3]Research Assistant (Botany),
[4]Assistant Director (Siddha)
Siddha Regional Research Institute, Poojappura, Thiruvananthapuram, Karala.

Email: vglatha@gmail.com

ABSTRACT

The present study is intended to characterize Ayakantha chenduram, an important Siddha herbo-mineral formulation. Various characterization techniques like Fourier transform infrared spectroscopy (FTIR), Raman Spectroscopy, X – ray diffraction (XRD) and Scanning Electron Microscope (SEM) with Energy Dispersive Analysis of X-ray (EDAX) were carried out. Evidences were established to ensure the identity of Ayakantha 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. The X-ray diffraction studies identified that the major crystalline materials present in the formulation are α-iron oxide and γ-iron oxide. FT-IR and Raman spectral data are also supported the presence of iron oxide in the chenduram. The SEM-EDAX study gave important clues regarding the morphology and inorganic composition of the sample. Particles in nano range were identified. Further, the results obtained from the SEM analysis show that most of the particles present in Ayakantha chenduram are in nano and near nano size. The data retrieved from the current characterization study reveals the size, shape and nature of the components present in Ayakantha chenduram.

Keywords: Ayakantha chenduram, XRD, SEM, FTIR

INTRODUCTION

Indigenous systems of medicine are believed to be one of the most ancient and well organized traditional health-care for rural as well as urban populations in India. But one of the major bottlenecks in the wider acceptance of herbal drugs from developing countries is the inadequacy or lack of standardization which embodies total information and controls that are necessary to guarantee the consistency of composition of the product ensuring its quality. In Siddha system, minerals and metals are used in addition to plant and animal constituents. Apart from gold and silver, mercury, sulphur, mica, arsenic, zinc etc. are treated with other single drugs and are given as bhasmas and chendurams. The Siddha practitioners assert that even though the metal in elemental form is toxic, in compound form they are human-safe. But the need of the hour is to bring out the importance of Siddha system worldwide by ensuring its

quality, efficacy and safety. Due to the advancement of scientific technologies, standardization techniques for checking the quality of these medicines to meet the criteria to support it worldwide become feasible. Hence the importance of some of the modern state-of-the-art techniques such as FT-IR, Raman Spectroscopy, XRD and SEM with EDAX are discussed in this paper.

According to Siddha system of medicine, Chenduram is a red coloured powder generally made of metallic compounds as major ingredients. This paper deals with the standardization of Ayakantha chenduram, a herbo-mineral Siddha medicine prepared as per the Siddha classical text by the process of calcination. Ayakantha chenduram is used in the treatment of various disorders especially Anaemia and Edema. Anaemia is a very common health problem found today, usually in females and children. As per modern science, Anaemia is not classified as a disease. But Siddha science has placed Anaemia not as a state or condition but a disease. Signs and symptoms of anaemia are tiredness on mild exertion, Nausea, Loss of appetite, Giddiness, Palpitation, Loss of general health and stamina, Loss of weight, Pallor of conjunctiva, Pallor and thickening of nail bed. Parpams and Chenduram like Aya chenduram, Kantha chenduram, Velli chenduram, Armugha chenduram, Ayakantha chenduram are good haematincs. Ayakantha chenduram is also used for the treatment of liver problems.

In this study an attempt is made to characterize Ayakantha chenduram in terms of its physico-chemical properties using modern techniques such as XRD, SEM with EDAX, FTIR and Raman spectral studies. The present study shows that the metals in this preparation are in sulphide form which enhances/confers the therapeutic efficacy of the drug. Particles in nano range were also identified.

MATERIALS AND METHODS

Ayakanthachenduram

The Siddha classical medicine Ayakantha chenduram was procured from IMCOPS (The Indian Medical Practitioner's Co-operative Pharmacy & Stores Ltd.). The drug was prepared by the methods of Agasthiyar chenduram – 300. Ayakantha chenduram is prepared using 12 ingredients which are (1) *Ayappoti* (Metallic iron powder), (2) *Kanthakam* (Sulphur), (3) *Kantham* (Lode stone), (4) *Linkam* (Cinnabar), (5) *Venkaram* (Borax), (6) *Patikaram* (Common alum), (7) *Puniru* (Alkaline earth salt), (8) *Corruppu* (Common salt), (9) *Intuppu* (Common salt), (10) *Navaccaram* (Sal ammoniac), (11) *Karpuram* (Camphor) and (12) *Elumiccam palaccaru* (Lime juice). Grind items 1 to 11 with item 12 for 2 days. Make cakes, dry and calcine using 100 cow dung cakes. Repeat the calcinations till it attains dark brown colour.

Organoleptic and Physical properties

The organoleptic characters such as colour, touch, taste and odour were noted.

Physico-chemical parameters

Ayakantha chenduram was subjected for the determination of physico-chemical parameters such as total ash, acid-insoluble ash and water soluble ash using standard methods.

Inorganic qualitative analysis

Inorganic qualitative analysis was carried out by standard laboratory methods.

FT-IRspectral analysis

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

Raman spectral analysis

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

XRD analysis

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

SEM and EDAX analysis

The elemental composition of the sample was determined Carl Zeiss, Germany Model: SUPRA

55VP, Gemini Column SEM with air lock system with EDAX Oxford Instruments X-MAX ($20mm^2$). A representative portion of each sample was sprinkled onto a double side carbon tape, then mounted on Aluminium stubs and sputtered, 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

The present study evaluated the physico-chemical characterization of nanosized and herbo-mineral Siddha formulation, Ayakantha chenduram. It is a fine dark brown powder. The chenduram was subjected to other physical tests and found that it was able to float on water which ensured its lightness, able to be inserted in furrows of finger of human hand which ensured particle size and freeness of particles from adhesives to each other.

The Loss on drying (LOD), total ash value, water soluble ash and acid insoluble ash were found to be 0.618, 97.28, 95.67 and 1.80% respectively. The results are consistent with those in Siddha Pharmacopeia standards. The LOD values were found to be low, as the moisture content and organic matter were removed during the process of calcinations[2]. Acid insoluble ash was found to be low, which may be due to the soluble nature of inorganic materials in acid medium[3].

The qualitative analysis showed the presence of Sodium, Aluminium, Silicon, Chloride, Sulphate, Potassium and Iron. Surface-volume ratio is an important criterion for assessing the nanotechnology application. Surface-volume ratio depends on the size and shape of the particles. It is therefore necessary to characterize the synthesized nanoparticles according to their size and shape. The synthesized nanoparticles were subjected to chemical characterization in order to validate the chemical property of nano iron and also to detect the involvement of molecules in the synthesis [4].

The FT-IR peaks indicated that Si-O bond and Fe-O bond formation exists in the compound and the peaks were well matched with reported FT-IR spectral of Iron oxide. The FT-IR spectrum of the product shows the presence of well-crystallized iron oxide nanoparticles. The IR bands show strong peak at 555 cm^{-1}. This peak may correspond to Fe-O stretching and bending vibration mode, of γ-Fe_2O_3[5]. This peak may correspond to a partial vacancy ordering in the octahedral positions in the maghemite inverse spinel crystal structure[6-8]. The characteristic absorption band at 466 cm^{-1} may be assigned to Fe-O stretching and bending vibration mode of α-Fe_2O_3 respectively [9].Typical peak at 1097 cm^{-1} was due to formation of alpha phase of iron oxide compound with siliceous-iron based compound. No peak at 2900 cm^{-1} indicating the absence of C – H stretching band which means all organic compounds are removed from the samples after calcinations at 700°C (Figure 1).

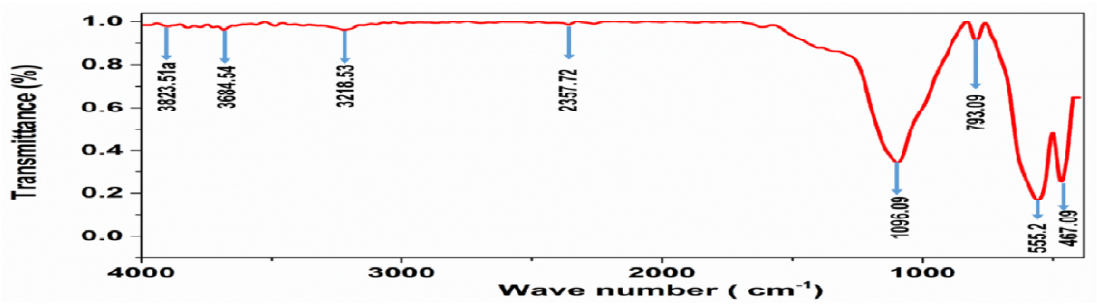


Fig.1: FT-IR spectrum of Ayakanthachenduram

Raman spectroscopy is a spectroscopic technique used to observe vibrational, rotational, and other low-frequency modes in a system. Raman spectroscopy is commonly used in chemistry to provide a structural fingerprint by which molecules can be identified. The Raman spectrum of the Ayakantha chenduram is presented in Fig. 2. In the range of 200–1750 cm^{-1}, many peaks could be observed, which were located at 218, 285, 404, 501, 617, 648, 1266, 1275, 1287, 1300, 1311, 1323, 1335, 1357, 1423, 1601, 3028, and 3061 cm−1, respectively. These peaks show the presence of α-Fe_2O_3 and γ-Fe_2O_3 phases. The Raman peaks appearing at 218 and 501 cm^{-1} were assigned to A1g mode, and those at 285, 404, and 617 cm^{-1} were assigned to Eg modes. The peak located at 648 cm^{-1} is attributed to disorder effects and/or the presence of Fe_2O_3 nanocrystals, while the peak observed at 1323 cm^{-1}has been assigned to hematite and 1423 $cm^{-1}$to maghemite [9-11].

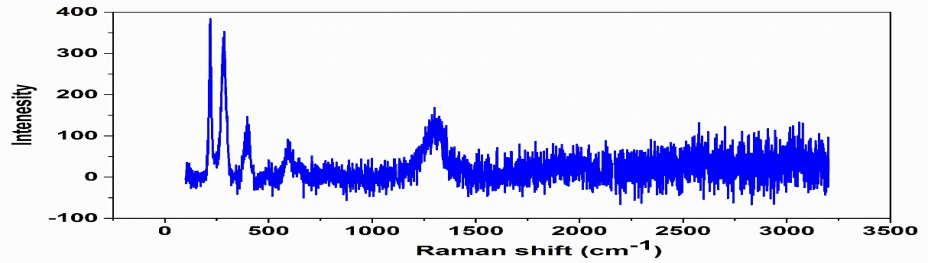


Figure 2: Raman spectrum of Ayakantha chenduram

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 Ayakantha 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/-039-1346). It can be seen from Fig. 3 that eight characteristic peaks observed for Ayakantha chenduram (2θ= 24.14º, 33.15, 35.61º, 40.85º, 49.48º, 54.16ºand 57.35º) can be attributed to the (012, 104, 110, 113, 024, 116, and 018) crystalline structures which corresponds to α – Fe_2O_3. The peaks observed at 26.1, 30.24, 43.28, 62.97 and 64.04 can be attributed to the (211, 220, 400, 440 and 441), crystalline structures which corresponds to pure γ – Fe_2O_3. The peaks can be indexed to a cubic spinel phase of γ- Fe_2O_3Maghemite, JCPDS No. 89-5892 and pure rhombohedral phase of α-Fe_2O_3Hematite, JCPDS No. 33-0664.

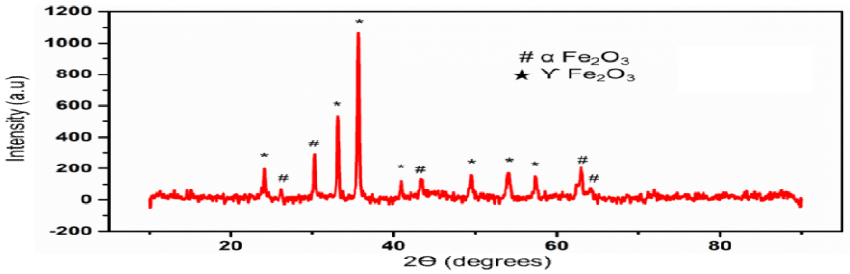


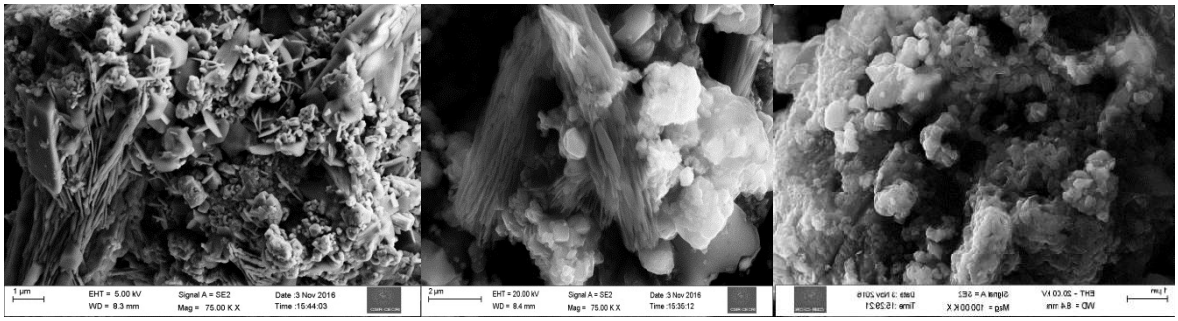
Figure 3: XRD pattern of Ayakanthachenduram

The materials showed good crystalline in nature with particle size of below 500 nm, which was also predicted and confirmed by SEM analysis. Elemental composition of Ayakanthachenduram was determined by EDAX analysis.

The Iron content was 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. The relation between siddha medicine and nanotechnology is mostly related to the size. The size of some siddha powders like Ayakantha chenduram, ayabringa chendrum are observed nearly 25 nm in size as per the investigation using AFM analysis. And the some of the nature of pashubams such as Ag, Au is similar to that of functionalized gold and silver quantum dots. It seems to be siddha medicine uses nanoparticles long years before.

The SEM images clearly indicated the presence of aggregated particle formation with alpha-iron oxide as a major phase. Agglomerations of particles are may be due to the repeated cycles of calcination and grinding involved in preparation. SEM can achieve resolution better than 1 nanometer[12]. The size of the particles is also less due to the repeated calcination and grinding[2,13]. 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 Ayakantha 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. SEM images showed the morphology of this formulation as nanorods. It is widely believed that formulation containing nanoparticles will effectively bind with the bacterial membrane and will aid in penetrating the cellular component of the organism thereby preventing its replication. It is also hypothesized that biomolecules and organic acids present in biological entities/ substrates can control the structure, phase, orientation and nanostructural topography of inorganic crystals, resulting in nanosized particles[10].



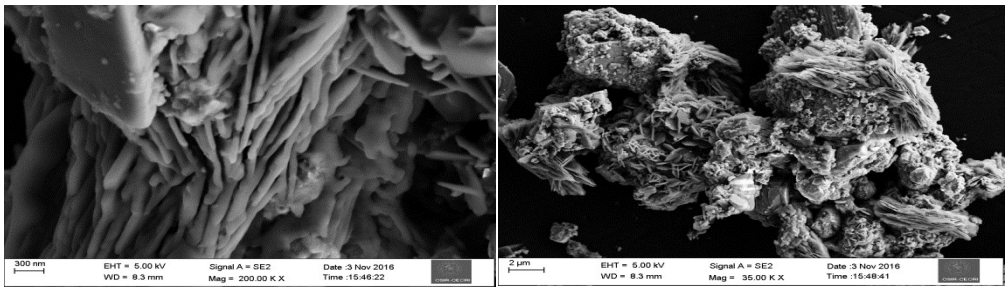


Figure 4: SEM pattern of Ayakanthachenduram

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. EDAX data showed the exact amount of respective element present in the sample in terms of wt % by wt%.

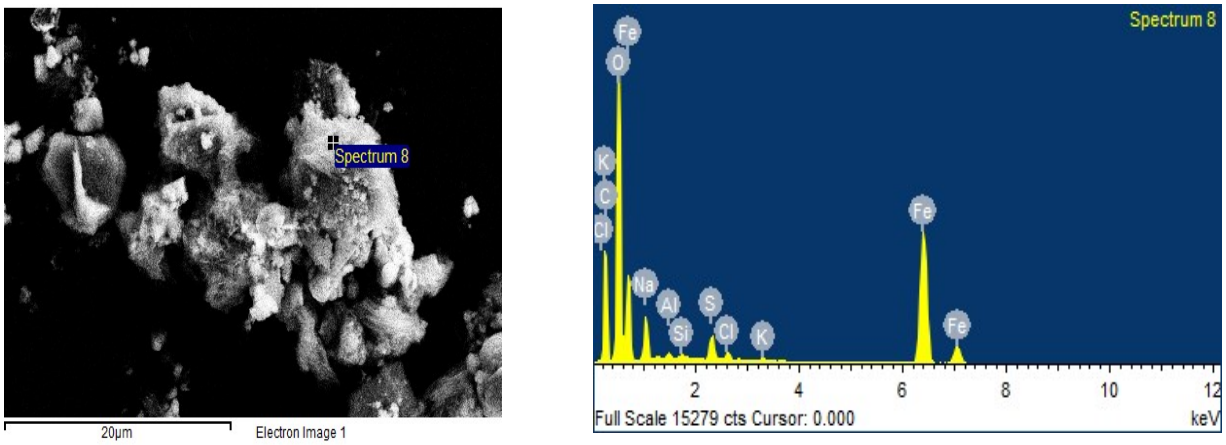


Figure 5a: Electron image of Ayakantha chenduram using SEM;
Figure 5b: Elemental composition of Ayakantha chenduram using EDAX

Table 1: Elemental composition of Ayakantha chenduram using EDAX

Sl. No.	Element	Weight%	Atomic%
1.	C (Carbon)	30.16±0.15	43.86±0.12
2.	O (Oxygen)	41.57±0.13	45.38±0.10
3.	Na (Sodium)	3.27±0.26	2.48±0.14
4.	Al (Aluminium)	0.29±0.02	0.19±0.003
5.	Si (Silicon)	0.14±0.01	0.09±0.47
6.	S (Sulphur)	1.02±0.05	0.56±0.02
7.	Cl (Chloride)	0.33±0.02	0.16±0.004
8.	K (Potassium)	0.11±0.05	0.05±0.27
9.	Fe (Iron)	23.10±0.16	7.22±0.11

The results of EDAX analysis are given in Table 1, 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 major element observed in the spectrum as a sharp signal. Along with that, some weak background signals of Si, Cl, K, etc., were observed which might have come from the debris on the EDAX chamber[8]. 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). Thus, the principle peak of Fe in the range of 6-8 Kev further validated the predominance of iron nanoparticles in this formulation.

CONCLUSION

The present study reveals that the heavy/ toxic metals As, Pb, Cd and Hg were found to be absent, which ensured the safety of this formulation. XRD studies confirmed the alpha iron oxide phase and gamma iron oxide phases existed in the formulation. SEM analysis shows the aggregated particle morphology for the formulation. The above investigation on Ayakantha chenduram using modern techniques establishes the fingerprint for standardization of the herbal formulation. In this study Ayakatha 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-Fe2O3.

ACKNOWLEDGEMENT

The authors are thankful to the Director General, Prof. Dr. R. S. Ramaswamy, Central Council for Research in Siddha, Chennai for providing necessary facilities to carry out this work.

REFERENCES

1. Siddha Formulary of India. Ministry of Health & Family Welfare. Govt. of India. 1992. Part I, Edn. I, pp. 37.
2. Rajalakshmi P, Devanathan R, Brindha P. Analytical studies on *MuthuchippiParpam*. J Pharm Res 2010; 3:2366-70.
3. Yadav S, Debnath M. Pharmaceutical and physico-chemical study of *KasisaBhasma*. Indian J Ethno-Phytopharm 2016; 2:43-9.
4. Subramaniyam, V., Subashchandrabose, S. R., Thavamani, P., Megharaj, M., Chen, Z., and Naidu, R. Chlorococcum sp. MM11—a novel phyco-nanofactory for the synthesis of iron nanoparticles. J. Appl. Phycol. 2015. *27*(5), 1861-1869.
5. Kim Il T., Nunnery G. A., Jacob K., Schwartz J., Liu X., and Tannenbaum R., Synthesis, characterization, and alignment of magnetic carbon nanotubes tethered with maghemite nanoparticles. J. Phys. Chem. 2010. 114 (15) 6944–6951.
6. White W.B., and Deangelis B.A.,. Interpretation of the vibrational spectra of spinels. Spectrochim.Acta, Part A Mol. Spectr. 1967. 23 (4), 985-995.
7. deFaria D.L.A., Silva, S.V., and de Oliveria M.T. Raman microspectroscopy of some iron oxides and oxyhydroxides. J. Raman Spectrosc. 1997. 28 (11), 873–878.
8. Millan A., Palacio F., Falqui A., Snoeck E., Serin V., Bhattacharjee A., Ksenofontov V., Gu¨tlich P., and Gilbert I. Maghemite polymer nanocomposites with modulated magnetic properties.Acta Mater. 2007. 55 (6) 2201–2209.
9. Zhao B., Wang Y., Guo, H., Wang J., He Y., Jiao Z., Wu M. Iron oxide(III) nanoparticles fabricated by electron beam irradiation method. Materials Sci Poland 2007. 25 (4) 1143-1148.

10. Ghashghaei S, Emtiazi G. Production of hydroxyapatite nanoparticles using tricalcium-phosphate by Alkanindigesillinoisensis. J Nanomat. Mol. Nanotechnol. 2013. 2:5
11. Khanra, K., Panja, S., Choudhuri, I., Chakraborty, A., and Bhattacharyya, N. Evaluation of antibacterial activity and cytotoxicity of green synthesized silver nanoparticles using Scopariadulcis. Nano Biomed. Eng. 2015. 7(3), 128–133.
12. Marrero, J., Polla, G., Rebagliati, R. J., Plá, R., Gómez, D., and Smichowski, P. (2007). Characterization and determination of 28 elements in fly ashes collected in a thermal power plant in Argentina using different instrumental techniques. *SpectrochimicaActa Part B: Atomic Spectroscopy*, *62*(2), 101-108.
13. Devanathan R, Rajalakshmi P, Brindha P. Chemical standardisation of *VaratikaBhasma*. Int J Cur Pharm Res 2010:2; 12-16.

Source of Support: Nil
Conflict Of Interest: None Declared

How to cite this URL: Gayathri Devi V et al: Characterisation Of Ayakantha Chenduram, A Nanosized Herbo-Mineral Formulation Using Modern Techniques. International Ayurvedic Medical Journal {online} 2018 {cited July, 2018} Available from: http://www.iamj.in/posts/images/upload/1343_1350.pdf