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ANALYTICAL STUDY OF HERBO-MINERAL PREPARATION-TAARA MANDOORA GUDA USING SEM-EDAX & XRD

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ABSTRACT

Analytical procedures are done to ensure the quality and standard of different formulations. There are different analytical methods for formulations containing herbal preparation, mineral preparation, herbo-mineral preparation etc., In the present study, the drug sample is subjected to SEM coupled with EDAX and XRD studies as the drug is a herbo-mineral preparation to know the elemental composition of the same. The drug Taaramandoora guda, is a herbo-mineral preparation, consisting of Mandoora which is indicated for the treatment of the Anaemia. The sample is prepared as per the ayurvedic texts, and it is subjected for the analytical study of the sample for the standardisation of the drug and to study the therapeutic utility of the same. The results showed that the particle size of the Mandoora is within the permissible limits and it is present in the oxide form which does not create any harmful effects in the human body. Traces of other elements were also found which is due to the other herbal ingredients in the sample. The study revealed the presence of compound that are beneficial for the human body and hence the classically prepared ayurvedic preparation does not pose any threat to the health of the human body.

KEYWORDS: Analytical study, SEM-EDAX, XRD, Taara Mandoora guda, Elemental analysis.

INTRODUCTION

The Analytical procedures provides the essential details regarding the constituents of the drug sample prepared, on the basis of which the pharmacodynamics of the drug can be understood in a better way and hence the improvement in the formulation of the drug can be done on this basis. The Scanning Electron Microscope (SEM) gives a detailed picture about the minute details like the particle size, the binding between the components in the formulation etc., when this is coupled with the Energy Dispersive X-Ray Spectroscopy (EDX/ EDAX), the elements present can be known along with its percentage i.e., the quantity of the element can be known accurately. X-Ray Diffraction (XRD) is another of analytical procedure which helps in the quantitative and qualitative analysis of the drugs especially the mineral drug present in the sample. With the help of this procedure, we can determine the element present in the sample in its compound form, the bonding of the elements and its structural form along with its percentage in the sample.

In this study, the procedure of SEM-EDAX and XRD is for the analysis of the herbo-mineral drug Taaramandoora Guda (TMG) an ayurvedic formulation as sample.

MATERIALS AND METHODS

Centre for Study: Central Instrumentation Facility, MAHE, Manipal.

Composition of the drug Taaramandoora guda[1]

The formulation contains Vidanga (Embelia ribes), Chitraka (Plumbago zeylanica), Chavya (Piper chaba), Triphala (Terminalia chebula, Terminalia bellerica, Emblica officinalis), Trikatu (Piper longum, Piper nigrum, Zingiber officinale) each 1 part, 9 parts of Mandoora Bhasma (calx of Iron rust), 9 parts of Jaggery and 18 parts of Cow's urine.

Preparation of the drug sample for Analysis

- SEM-EDAX- The sample Taaramandoora guda (TMG) pills, is prepared with the dimension of 2cm diameter. The dried pill of TMG was sputtered with the silver to be subjected for the analysis.
- XRD- The sample of TMG prepared according to the specified dimension for pellets- 2mm thickness and 2 cm diameter. This sample was placed in the sample holding slot of the instrument for analysis.

Principle

SEM-EDAX[2]: The Scanning electron microscope works on the principle of applying kinetic energy to produce signals on the interaction of the electrons. These electrons are secondary electrons, backscattered electrons and diffracted backscattered electrons which are used to view crystallized elements and photons. Secondary and backscattered electrons are used to produce an image. The secondary electrons are emitted from the specimen play the primary role of detecting the morphology and topography of the specimen while the backscattered electrons show contrast in the composition of the elements of the specimen.

XRD[3]: XRD analysis is based on constructive interference of monochromatic X-rays and a crystalline sample: The X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda=2d \sin \theta$). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample.

The characteristic x-ray diffraction pattern generated in a typical XRD analysis provides a unique "fingerprint" of the crystals present in the sample. When properly interpreted, by comparison with standard reference patterns and measurements, this fingerprint allows identification of the crystalline form.

Description of Instrument[4]

- **SEM**
  - EVO MA18 with Oxford EDS(X-act)
  - Magnification: Minimum 1x and maximum 1,00,000x
  - EDS: Liquid nitrogen free
  - Sample holders: 9 sample holders
  - Depth of focus: At a magnification of 1000x, aperture size of 100 microns and working distance of 10 mm should be 40 microns.
  - Air conditioned (21 to 24 degree C); Relative humidity is less than 60%; Nitrogen with 99.99% purity, regulator (0-3 bar control)

- **XRD**
  - Rigaku Miniflex 600 (5th gen)

- X-ray generation – up to 40 kV & current (15 mA)
- Temperature range - near room temperature measurement with an up gradation of high temperature measurement
- Samples – Both bulk and thin films sample measurement.
- Filters – Nickel filter.
- Wide angle measurement (50-130°) 2 theta value.

Procedure

The prepared samples of the specified dimensions are placed in the sample slots of respective instrument and the procedure is started.

In SEM-EDAX, the area to be visualised can be changed using the controls in the instrument. The images are expressed after clear tropical image of required power can be obtained looking at the image over screen. With EDAX, the elements that are to be verified for its presence in the sample can be checked. The element present and its percentage is assessed by the instrument and report in the form of graphical presentation is prepared.

In XRD analysis, the incident X-Rays are directed towards the sample with an angle 2θ, the graphical presentation of the emitted rays from sample is captured by detector and presented on the monitor. The suspected element is tracked in the sample, and the compounds of those elements are detected by comparing it with the Standard graphical presentation of compound and results are interpreted.

RESULTS

In the SEM-EDAX, the following elements were detected in the targeted surface of the test sample TMG as given in Table.1. The particle size was found to be In powder microscopy, particle size was identified between 0.62µm-258µm.

Table 1: SEM-EDAX report of the sample TMG.

Element	Weight%	Atomic%
C	33.79	50.46
N	10.96	14.03
O	19.21	21.54
Mg	1.00	0.74
Al	0.75	0.50
Si	3.32	2.12
S	0.68	0.38
Cl	2.27	1.15

K	2.44	1.12
Ca	1.49	0.67
Mn	1.08	0.35
Fe	20.01	6.43
Cu	0.46	0.13
Zn	0.28	0.08
Cd	1.54	0.25
Hg	0.34	0.03
Pb	0.37	0.03
Totals	100.00	

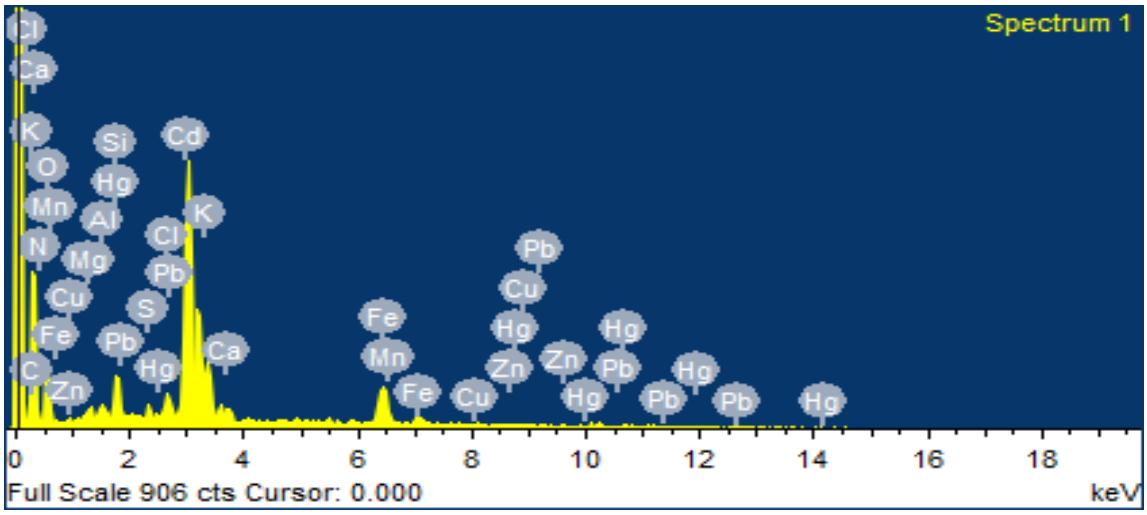

Figure 1: Graphical presentation of elements from sample using EDAX.

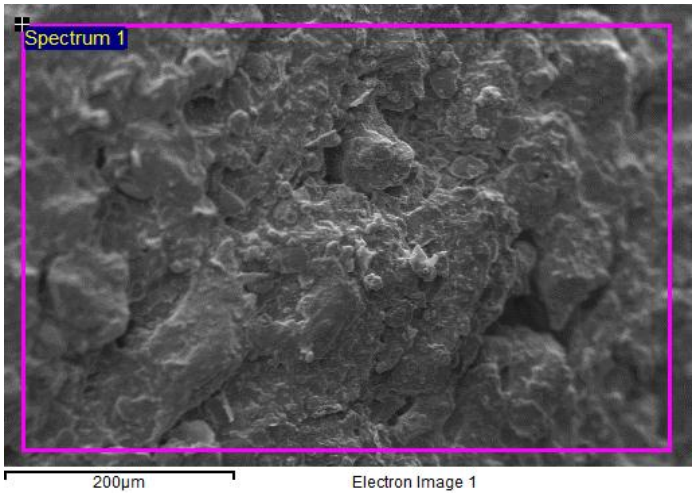

Figure 2: SEM Images of TMG.

Crystallographic study by XRD

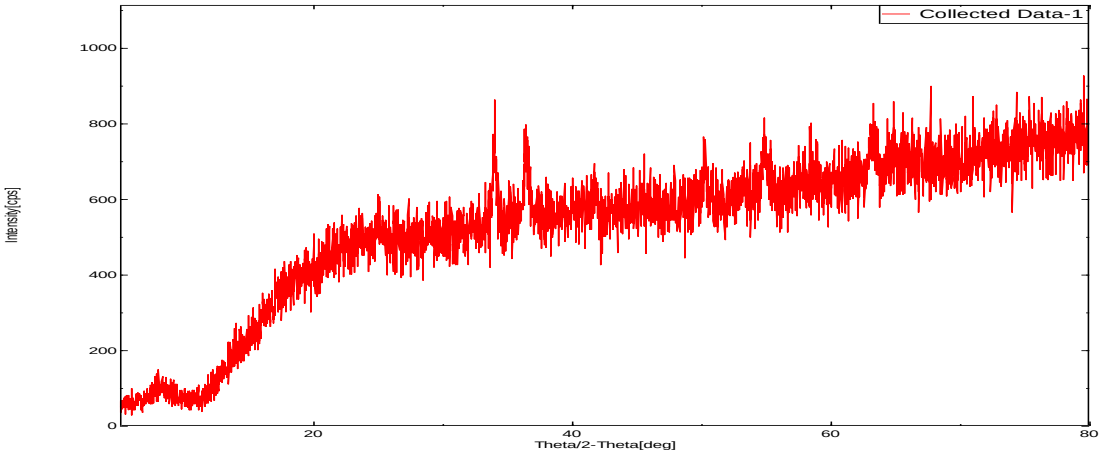

Figure 3: Graphical representation of intensity of scattered x-ray from the sample TMG.

The X-ray spectrum obtained was compared to the spectrum of Fe, FeO, Fe_2O_3, Zn, Ca, S, Cl, C, O, Mg, Mn for their presence. The tentative assignment was made to the intense peak appearing at various 2θ values.

The prominent peaks correspond to Fe_2O_3 with 2θ values 36.495, 60.899 and 63.206 indicating the presence of iron in the form of ferric oxide with rhomboid crystal structure presenting as Haematite.

Weak signals appearing at 2θ values 32.79, 55.33 was assigned to the $MnSO_4$, 2θ= 21.29 to $ZnSO_4$, 17.24 to $ZnCl_2$, 25.44, 24.85 corresponding to the $CaSO_4$, $CaCO_3$ respectively. And few signals corresponding to $MgSO_4$ with 2θ value 24.3. XRD study indicated the presence of Fe_2O_3, traces of Zn, Mn, Mg, Ca and almost absence of heavy metals in free form.

DISCUSSION[5]

The drug TMG was subjected for elemental analysis, for this purpose the SEM-EDAX method was chosen. In the Scanning Electron Microscope, coupled with the Energy Dispersive X-ray Analyzer provides detailed high-resolution images of the sample by rastering a focussed electron beam across the surface and detecting secondary or backscattering electron signal. EDAX is also used to provide elemental identification and quantitative compositional information. The results of the analysis of sample TMG confirmed the presence of organic elements Carbon, Oxygen, Nitrogen along with Iron, Silicon, Potassium, Chlorine, Cadmium, Calcium, Manganese, and Magnesium in descending order

of percentage composition with traces of Al, Zn, Cu, S, Pb and Hg. The Hg and Pb are in negligible amount as given in the Table 1.

The X-Ray Diffraction is a rapid analytical technique primarily used for the phase identification of the crystalline material and to provide information regarding the chemical composition of the sample analyzed. The X-ray spectrum obtained was compared to the spectrum of Fe, O, Cu, Mn, Mg, Cl, Zn, S for their presence. The prominent peak corresponds to the iron oxide indicated the presence in the form of ferric oxide Fe_2O_3, resembling crystallography of hematite.

- The Taaramandoora guda sample consisted of elements like- Carbon, Nitrogen, Oxygen, Iron, Silica, Chloride, Potassium, Cadmium, Calcium, Manganese and Magnesium along with other trace elements. The heavy metals were detected but it was under the permissible limits as per IP.
- The interpretation of the graph of the drug TMG, the majority of the peaks coincided with the peaks of iron oxide and by the observation of the angle of deflection of the rays in the graph it can be inferred that, the majority of the compounds present in the sample is Iron Oxide with the rhomboid crystal structure imitating the crystal structure of Hematite, along with weak peaks of magnesium sulphate, zinc sulphate, zinc chloride, calcium sulphate, calcium carbonate, zinc oxide, magnesium sulphate suggesting the presence of these compounds in traces.

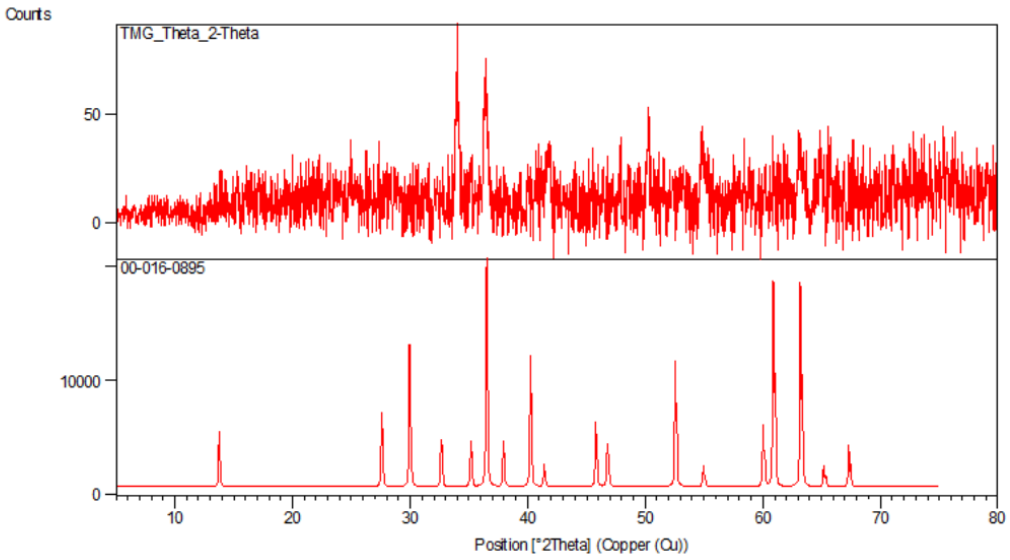

Figure 4: Determination of peaks from the XRD report of sample TMG.

CONCLUSION

The elemental analysis of the Taaramandoora guda confirms the presence of Iron oxide and traces of compounds like- magnesium sulphate, zinc sulphate, zinc chloride, calcium sulphate, calcium carbonate, zinc oxide, magnesium sulphate.

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