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ANALYTICAL STUDY OF CHINCHA KSHARA THROUGH MODERN PARAMETERS LIKE XRD, SEM AND EDS

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ABSTRACT: Analytical study of Ayurvedic preparations is the need of present scientific era. Though the Ayurvedic drugs are time tested and have been used successfully in the management of various ailments, it is now necessary to prove their quality, efficacy and safety to the scientific world through various modern analytical parameters. Chincha kshara is an important preparation mentioned in ayurvedic classics for the management of various diseases. An attempt has been made in the present study to prepare Chincha kshara and analyse the physical nature, surface morphology and chemical constituents present in the Chincha kshara through analytical parameters like organoleptic properties, pH value, X-ray diffraction technique (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). XRD study of Chincha kshara showed the presence of potassium carbonate hydrate as major and minor peaks and sodium chloride, potassium calcium hydrogen carbonate phosphate hydrate, silver phosphate and magnesium phosphate oxide as minor peaks. SEM revealed its amorphous nature and EDS reported the significant presence of Potassium (50.44%) and Oxygen (46.07%) in Chincha kshara.

INTRODUCTION: Analytical study plays an important role in the standardization of the drugs. Ayurveda, the ancient system of medicine is gaining recognition throughout the World and many herbal, metallic and mineral drugs are now clinically tested and accepted. However, one of the impediments in the acceptance of these medicines is the lack of standard quality control profiles. The quality of the drug that is the profile of the constituents in the final product has implication on its efficacy and safety.

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Due to the complex nature and inherent variability of the chemical constituents of these drugs (herbal, metallic and mineral), it is difficult to establish their quality control parameters. Therefore modern analytical techniques are expected to help in circumventing this problem. Hence highly sensitive modern parameters are employed for gaining information about identity, form, surface morphology and structure of contents of the formulation.

Kshara are alkaline substances obtained by processing the ash of drugs. Chincha kshara is considered one among the Kshara ashtaka[1]. Acharya Charaka [2], Susrutha [3] and Vagbhata [4] described the properties of Kshara in detail. Detailed description regarding the preparation of various types of Kshara and their properties has been mentioned under Kshara kalpana. Kshara are considered as Anushastras because they do

chedana, bhedhana, lekhana *etc*. actions which are possible by using sharp instruments (Shastras) [5]. Chincha kshara is prepared from Chincha Phala twak or Kanda twak. Chincha kshara possesses rich medicinal values and is used as an important ingredient in various formulations which are used in the management of Shula, Agnimandya, Gulma, Mutrakricchra, Ashmari *etc* [6].

In the present study, Chincha kshara was prepared according to the reference mentioned in Sarangadhara Samhitha11/102-104 and it was subjected to different analytical tests like organoleptic properties, pH value, XRD, SEM and EDS for the identification of crystalline materials, surface morphology and chemical composition.

MATERIALS AND METHODS:

Procurement of Raw Materials: Dried Chincha phala twak was collected from Adilabad district of Telangana. Authentication was done on the basis of pharmacognostical characters.

Pharmaceutical Study:

Preparation of Chincha kshara:

- Chincha phala twak was collected and completely dried. It was placed over an iron mesh. Then it was placed over the hearth and subjected to fire till it gets converted into ash.
- The ash was collected in a stainless steel vessel and allowed for self-cooling.
- Four parts of water was added to the ash obtained. It was kept undisturbed overnight. Then the supernatant water was collected in another steel vessel carefully without allowing the sediments to enter.
- The collected supernatant water was heated under medium flame on a gas stove till the water content was completely evaporated.
- After the complete evaporation of water content, white coloured flakes (kshara) were obtained at the bottom of the vessel.They were pounded in Khalwa yantra and made into fine powder. (**Table 1**)
- Chincha kshara was collected and preserved in air tight glass container.

Analytical Study:

Organoleptic Properties: Chincha kshara was tested for taste, odour, colour, appearance and touch.

pH Value: pH test was carried out to know the nature of Chincha kshara.

X-ray Diffraction Technique: X-ray Diffraction was Performed to identify the crystalline materials present in Chincha kshara. 1 g of Chincha kshara was taken and powdered in agate mortar to very fine powder. It was mounted in a sample tray of machine. X-ray beam bearing a wavelength of 1.5418740 Å from copper source is passed on the sample. Detector was set to identify diffracted beams between 10 - 70 degrees of 2 range. Obtained values were plotted on graph with the help of inbuilt "Reyflex Software" for further analysis.

Scanning Electron Microscopy: Scanning electron microscopy (SEM) was conducted to know the surface morphology of Chincha kshara. Sample of Chincha kshara was analyzed directly by keeping it on the specimen holder and observed under the scanning electron microscope at 1,000 KX - 10,000 KX magnifications. Micrographs were taken with the inbuilt camera.

Energy Dispersive X-ray Spectroscopy: EDS was performed to know the chemical composition of Chincha kshara. Sample of Chincha kshara was placed on the specimen holder and subjected to Energy-Dispersive X-ray spectroscopy (EDS). When the sample is bombarded by the SEM's electron beam, electrons are ejected from the atoms comprising the sample's surface. The resulting electron vacancies are filled by electrons from a higher state and an X-ray is emitted to balance the energy difference between the two electrons' states. The X-ray energy is characteristic of the element from which it was emitted.

RESULTS:

TABLE 1: SHOWING RESULTS OF PHARMACEUTICAL STUDY

Weight of Chincha phala twak	Weight of the ash obtained	Weight of Chincha kshara
28 kg	1085 g	350 g

TABLE 2: SHOWING ORGANOLEPTIC PROPERTIES

S. no.	Parameters	Result
1.	Colour	White
2.	Odour	Characteristic odour
3.	Appearance	Fine powder
4.	Touch	Soft
6.	Taste	Salty

pH value of Chincha kshara is 13.

TABLE 3: SHOWING THE DETAILS OF MATCHING PEAKS OF XRD DATA FOR CHINCHA KSHARA

S. no.	Element/Molecule	JCPDS Ref. no.	2θ	Intensity	FWHM	H	K	L
1.	$K_2CO_3(H_2O)_{1.5}$ (Potassium carbonate hydrate)	01-073-0470	30.71	15.8	0.12	2	2	1
			32.62	100	0.12	3	3	0
			34.80	2.9	0.192	-4	2	2
			29.73	40.8	0.24	-3	3	1
			30.71	15.8	0.072	2	2	1
			38.50	17.5	0.12	1	5	1
			40.48	13.3	0.24	-2	2	3
			42.43	4.8	0.193	-2	6	1
2.	NaCl (Sodium Chloride)	00-005-0628	31.69	100	0.192	2	0	0
3.	$Ca_8H_2(PO_4)_6.H_2O.KHCO_3.H_2O$ (Potassium calcium hydrogen Carbonate phosphate hydrate)	00-047-0260	25.80	70	0.192	0	0	2
4.	$Ag_4P_2O_7$ (Silver phosphate)	00-037-0187	32.38	100	0.144	1	1	12
5.	KCl (Potassium chloride)	01-075-0296	28.34	100	0.192	2	0	0
6.	$Mg_2P_2O_7$ (Magnesium phosphate oxide)	00-005-0579	48.65	4	0.12	-	-	-

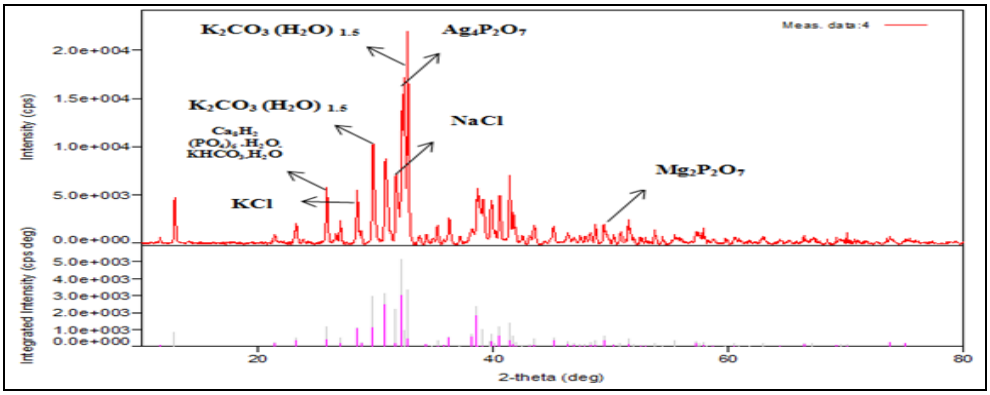

FIG. 1: SHOWING XRD OF CHINCHA KSHARA

XRD of Chincha kshara shows $K_2CO_3(H_2O)_{1.5}$ (Potassium carbonate hydrate) compound with monoclinic structure as major and minor peaks and NaCl (Sodium chloride) compound with cubic structure, $Ca_8H_2(PO_4)_6.H_2O.KHCO_3.H_2O$ (Potassium calcium hydrogen carbonatse phosphate hydrate) compound with hexagonal structure, $Ag_4P_2O_7$ (Silver phosphate) compound with hexagonal structure, KCl (Potassium chloride) compound with cubic structure and $Mg_2P_2O_7$ (Magnesium phosphate oxide) compound as minor peaks.

$K_2CO_3(H_2O)_{1.5}$ peaks were detected at diffraction angle of 30.71, 32.62, 34.80, 29.73, 30.71, 38.50, 40.48 and 42.43. NaCl peak was detected at diffraction angle of 31.69, $Ca_8H_2(PO_4)_6.H_2O.KHCO_3.H_2O$ at 25.80, $Ag_4P_2O_7$ at 32.38, KCl at 28.34 and $Mg_2P_2O_7$ at diffraction angle of 48.65.

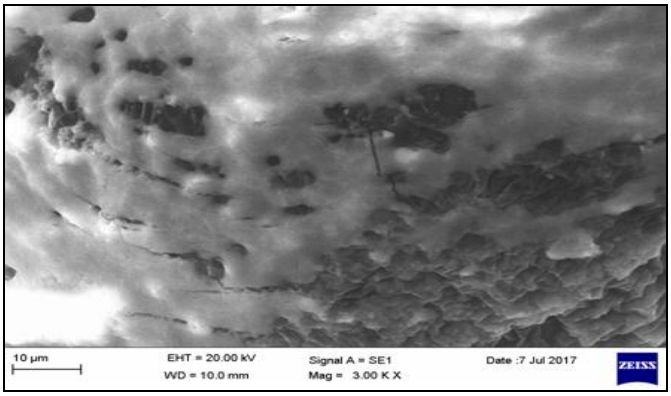

FIG. 2: SHOWING SEM RESULT OF CHINCHA KSHARA (MAG. 3KX)

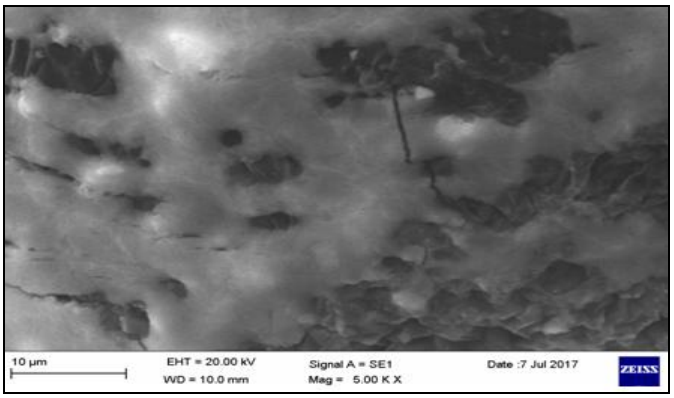

FIG. 3: SHOWING SEM RESULT OF CHINCHA KSHARA (MAG. 5KX)

SEM micrographs show the amorphous nature of particles of Chincha kshara with porous morphology at 3KX and 5KX magnifications. There was no particular pattern in the structure. Foamy appearance of particles of Chincha kshara was seen in the micrographs

TABLE 4: SHOWING THE QUANTITY OF THE ELEMENTS IN CHINCHA KSHARA

Element	Weight %
O K	46.07
Na K	0.31
S K	0.76
Cl K	2.42
K K	50.44
Total	100.00

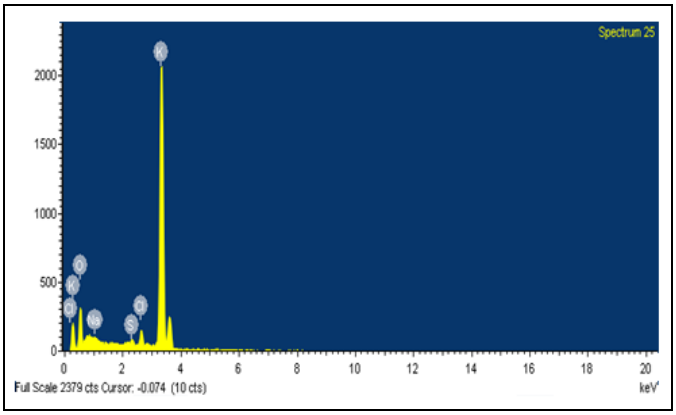

FIG. 4: SHOWING EDS OF CHINCHA KSHARA

EDS of Chincha kshara revealed the presence of Potassium (50.44%), Oxygen (46.07%), Chlorine (2.42%), Sulphur (0.76%) and Sodium (0.31%).

DISCUSSION: Kshara kalpana is an important dosage form described in various authoritative texts of Ayurveda. Kshara are obtained from the drugs of animal, mineral and plant origin. It is mentioned in the classics, that the diseases which are difficult to treat can be cured by Kshara therapy [7]. Due to the inherent properties present in Kshara they are extensively used in the management of various diseases. Kshara play a very important role in surgical procedures in the form of Kshara sutra for the management of ano-rectal diseases like piles, fistula *etc* [8]. In the recent era application of Kshara sutra in ano-rectal disease has become a common practice in ayurvedic surgical parlance. Various Kshara were used in number of Ayurvedic preparations but the elemental composition of Kshara still remained less studied. So, to throw adequate light on the analytical aspects of Chincha kshara this study has been conducted. Organoleptic properties of Chincha kshara are white colour fine powder with salty taste having characteristic odour and soft to touch. (**Table 2**)

pH value indicates the degree of acidity or alkalinity of a sample. pH value of Chincha kshara is 13, which indicates its high alkaline nature. Alkalinity of the drug indicates the site of absorption and action of the drug.

XRD is a rapid analytical technique used for the phase identification of crystalline materials and provides information on unit cell dimensions. It gives the fingerprint characterization of crystalline materials and determination of their structure. Each crystalline solid has its unique characteristic X-ray powder pattern, which may be used as a "fingerprint" for its identification. Size and the shape of the unit cell for any compound can be detected most easily using the diffraction of X-rays. XRD of Chincha kshara showed its crystalline nature. The predominant peaks in the sample corresponded to major phase comprising K_2CO_3 $(H_2O)_{1.5}$ (Potassium carbonate hydrate). Single minor peak of Sodium chloride, Potassium calcium hydrogen carbonate phosphate hydrate, Silver phosphate, potassium chloride and Magnesium phosphate oxide were observed. (**Table 3**) (**Fig.1**)

Potassium carbonate is a white salt, soluble in water which forms a strong alkali solution. Many major and minor peaks of potassium carbonate hydrate were seen along with other potassium containing compounds. This indicated the presence of potassium in significant amount in Chincha kshara.

SEM is an analytical technique that uses electron beam rather than light to form a figure. It is capable of producing high resolution figures of a sample surface, which means that closely spaced features can be examined at a high magnification. Due to the manner in which the fig is created. SEM Fig. have a characteristic three dimensional appearance and are useful for determining the surface structure of the sample. It can magnify objects to extreme levels where even structure of nano particles could be clearly visible. SEM micrographs of Chincha kshara depicted its amorphous nature and porous morphology. Particles were adhered together as agglomerates.

There was no definite pattern in their structure. This may be due to hygroscopic nature of Kshara. (**Fig. 2** and **3**) EDS is an analytical technique used for elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of X-ray excitation and a sample.

This analysis confirmed the significant presence of elements like potassium and oxygen. Chlorine was present in trace amounts. Presence of oxygen in higher amounts in Chincha kshara may be due to the absorption or adsorption of environmental oxygen by its hygroscopic nature. (**Table 4**) (**Fig. 4**)

X-ray diffraction technique and Energy dispersive X-ray spectroscopy showed the presence of potassium containing compounds and Potassium in significant amounts respectively. This may be due the presence of potassium in higher quantities in *Tamarindus indica* (Chincha). Research studies conducted to know the minerals present in Tamarind fruit and seed have proved the presence of potassium, calcium, sodium, magnesium, iron, zinc and phosphorous. Of all the minerals potassium is the element in highest concentration [9]. This justifies the presence of potassium in Chincha Kshara. The high concentration of potassium is nutritionally significant by playing a key role in Neuro-muscular function [10]. Moreover, potassium is considered as one of the alkali metals. All the alkali metals have single valence electron in the outer electron shell, which is easily removed to create an ion with a positive charge - a cation, which combines with anions to form salts. Potassium in nature occurs only in ionic salts. All the alkaline metals react violently with water, releasing hydrogen and forming hydroxides. All alkali hydroxides are very corrosive being strongly alkaline.

CONCLUSION: From the present study, it can be concluded that Chincha kshara has high alkaline nature due to the presence of elements like potassium and oxygen in significant amounts. The result obtained from these analytical tests, only re-establishes the fact explained by our Acharyas regarding the properties of kshara. This echoes the vast knowledge and deep insight of our Acharyas in designing Kshara kalpana as a separate dosage form. The current observations can be considered as a lead for further studies on other Kshara.

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CONFLICT OF INTEREST: No conflict of interest.

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