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RESEARCH ARTICLE

Synthesis and Characterization of Mandur Bhasma

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ABSTRACT

The present study deals with the synthesis and characterization of Mandur Bhasma. The synthesized bhasma was subjected to various bhasma pariksha described in Ayurveda including NPST to know the changes during the preparation. The characterization of prepared bhasma was also done by using modern analytical techniques such as EDAX, SEM, XRD, FTIR, BET which shows that mandur bhasma mainly consists of iron oxide. Elemental analysis confirms that along with Iron other essential trace elements are also present in the prepared Bhasma.

Key words: Bhasma, Mandur, Shodhan, Ayurveda, Triphala decoction.

1. INTRODUCTION:

Bhasma is multiingredient herbo- mineral or herbometallic formulation which can be used in various treatments. Metallic Bhasma is well known for its quick effectiveness, smaller dose and long shelf life. If these bhasma are not prepared properly they can be toxic to human body Hence, Bhasma Pariksha is important in Ayurveda to confirm well prepared Bhasma [1]. The Revalidation of Pharmaceutical procedures used for the preparation of Ayurvedic bhasma is essential as the variation in standard procedure and use of different raw materials causes alteration in final bhasma product.

Iron deficiency (anemia) is a common problem in the developing countries like India. The treatment of iron deficiency in the contemporary science results in several adverse effects viz. severe gastrointestinal irritation, necrosis of the mucous membrane, cardiovascular collapse; severe damage to brain and liver or even death might occur [2]. Hence, the better alternative such as Mandura bhasma is useful in such treatments. It is an iron based preparation which is useful in therapeutics of anemia, jaundice, poor digestion, edema, skin diseases etc [3]. In Ayurvedic literatures Mandur is known by the names as lohkitta, malayas, ayomala etc. Chemically it is ferrous ferric carbon dioxide and oxygen in which 2 atoms of iron are combined with 3 atoms of oxygen [4].

In the present study the mandur bhasma is prepared according to the method mentioned in Ayurvedic literature (Rasatarangini).The prepared bhasma was characterized by traditional Ayurvedic method and by using various analytical techniques such as EDAX-SEM, XRD, FTIR and BET. The NPST test also performed for confirmation of complete synthesis of Bhasma. The Numburi Phased Spot Test (NPST), a spot test based on a chemical reaction, a new technique for assessing the quality of prepared bhasma. When a drop of clear solution of substance under examination (Bhasma) is put on specially prepared chemical reacting paper, a spot appears which shows a series of color and pattern changes. This involves observations of the spot and its colour at successive phases spread over different time intervals. This technique was developed and standerdized by Dr. Namburi Hanumant Rao in 1970, it has been accepted by CCRAS, New Delhi [5]. Outcomes of this study could be used as standards for evaluating quality and reproducibility of the *Mandura bhasma*. The attempt has been made in present work to bring forth the physicochemical characterization as a step forward to standardization of Mandur bhasma.

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2. MATERIALS AND METHODS

Bhasmas are traditional Indian medicinal preparation in which metals undergo thermal treatment to bring about thermal decomposition, phase transition, Purification and Detoxification to convert into digestible oxides.

Mandur bhasma was prepared according to the Process listed in the Rasa Tarangini [6], and API [7] by giving following treatments

2.1 Shodhan (Purification):

It is the process of Purification and detoxification by which physical and chemical impurities and toxic materials are eliminated and substances are subjected for further processing. The shodhan process mentioned in the Rasa text is not only a process of chemical purification but also process of addition and separation which causes Physical, Chemical and Biological changes in the metal. These changes depend on the structure, constituents, impurity and properties of particular substance [8].

The raw material was subjected for following treatment

2.1.1Samanya shodhan:

The raw material used for the preparation of Mandur bhasma was heated till red hot and dipped in 5 different liquid media such as sesame oil, butter milk, cow urine, Kanji and Horse gram decoction respectively. The process was repeated 7 times in each liquid media.

2.1.2Vishesh Shodhan:

The samanya shodhita sample was then subjected for vishesh shodhan. This was carried out using triphala decoction. The samanya shodhita Mandur was heated to red hot and then immersed in the triphala decoction.

[Trihala Decoction: 1 part of Triphala and 8 part of water was taken and heated till it reduced to ¼ th of original quantity.]

After vishesh shodhan sample was subjected for Bhavan Process.

2.2 Bhavan:

It is the process of wet grinding in which shodhita Bhasma are ground with particular liquid media for specific period.The Bhavan was done using Bhanupak Process.

2.2.1Bhanupak:

The shodhita Bhasma was mixed with 400 ml of Triphala decoction and kept under sun light till complete evaporation of liquid. For getting more brittle and fine particles, on complete drying of mixture again 100 ml decoction of Triphala was added and dried under sun light. This process was repeated 7 times.

2.3 Maran (Incineratin):

The levigated doughy mass was subjected to heating in electric muffle furnace as well as by using traditional Putas. In this process the purified material was triturated with cow urine and triphala decoction using mortar and pestle. Then uniform pellets / coin shape (Chakrikas) were prepared and then kept between earthen pot (sharav), which was sealed with clay. The flat disc shape of pellets facilitates maximum exposure of heat during incineration (**Fig 1**).

The earthen pot heated using electric muffle furnace as well as using traditional puta system for comparative study. The sample was kept for slow heating for 1 ½ hr, then at constant temperature 650^0C for 3hrs.The temperature of Electric Muffle Furnace was given on the basis of temperature required for maran of material reported in the literature.

Incineration (Maran) by traditional method was achieved by using seven Ardhgaja Puta. As per classical text Rasaratna samuchhaya, the dimension of pit required for Ardhagaja Puta 45.3cm (l×b×h).The cow dung cake 250 in number were used for 1 Ardhagajaput (**Fig 2**).

The Mandur bhasma prepared after incineration (**Fig 3**) was then subjected for Physical and chemical characterization.

2.4 Physical Characterization:

The Ayurvedic texts also given some test on quality control of finished product. Different parameters like Lusterless, Rekhapurnatvam (particle size enters in the furrows of finger),Varitara(floating of product on water), Nirutha(inability to ragain metallic form), were studied.

Other physicochemichemical characteristics also performed such as NPST Test, Total ash content, acid insoluble ash content.

2.5 NPST Test:

It is one of the qualitative tests described for various Ayurvedic preparations. NPST helps in identifying various samples of bhasmas. It depends upon the pattern of the spot, which develops after a specific chemical reaction. For this test 0.25 gm of Mandur bhasma was taken in the test tube and 0.5 ml HNO_3 was added. The sample was heated till the bottom appears red hot and then it was cooled. After that 5 N HNO_3 and 5% HCl, 0.5 ml each, were added and heated again with shaking. The solution was used for reaction with potassium Iodide paper after 40 hrs. of its preparation. The reacting solutions were shaked well for 2 hrs before it was treated with chemical reacting paper. 10% Potassium iodide paper was allowed to react with above solution for 40 hrs and 72 hrs.

2.6 Total Ash content:

1 gm of sample was accurately weighed and spread at the bottom of crucible and incinerated at 450^0C for 3 hrs. Heated sample was allowed cool naturally. The residue was weighed and total ash content was estimated.

2.7 Acid insoluble ash:

The residue from total ash estimation was boiled with 25 ml of dil. HCl for 5 min. The insoluble matters wash with hot water transferred to a crucible, dried and weighed. The acid insoluble content was estimated.

2.8 Analytical techniques used for Characterization:

The Quality assurance of such medicines can be achieved only through good manufacturing practices, regulatory control, research and Physicochemical characterization with reference to authentic drug formulation. The prepared Bhasma was analyzed using sophisticated instruments such as Fourier transform infrared spectrophotometer (FTIR), Scanning electron microscope with energy dispersive analysis of X-rays (SEM - EDAX) , X- ray diffractometer (XRD) and BET to determine organic moieties , particle size , to analyze the surface area , to determine the elemental composition, to study morphology ,to determine the crystalline Phase and surface area respectively. The Details of instrument used for characterization of Bhasma are given bellow.

- X- ray diffractometer (XRD): The prepared Bhasma was characterized using X- ray diffractometer SHIMADZU AA -7000 ,equipped with photo scintillation detector , angular range $2\theta = 10 - 80^0$, rate of scanning 5^0 / min.
- Energy dispersive X- ray analysis (EDAX): The elemental analysis of final product at the end as well as during intermediate stages of its preparation was done using EDAX of Bruker XSHLASH-6 I30.
- Scanning electron microscopy (SEM): Fe-SEM of FEI – NovananoSEM-450 was used to study the morphology of bhasma at various stages of its preparation.
- Brauner Emmet Teller Method (BET): The surface area of prepared bhasma and raw bhasma were recorded using BET technique (QUANTACHROME AUTOSORB iQ -2).
- Fourier transforms Infrared spectroscopy (FTIR): The Fourier Transform Infra Red (FTIR) Spectra were recorded between 4000-1000 cm^{-1} in FTIR Spectrometer (SHIMADZU 8400).

3.0 RESULTS AND DISCUSSION

The final product prepared by traditional puta system and using Electric muffle furnace had been subjected to Ayurvedic tests viz. Lusterless, Rekhapurnatvam , Varitara, Nirutha. All these tests indicated preparation of proper Bhasma. [9].

The changes in weight of sample in Bhanupak Process and in Maran Process are shown in (**Fig 4 & Fig 5**) respectively.

The weight gain occurred in Bhanupak Process (Vishesh Shodhan) (Fig 4) is attributed to addition of Triphala decoction. While substantial weight loss observed in maran process (Fig 5) is due to combustion of Triphala. However, weight decreases gradually afterwards due to formation of iron oxide.

The color Change of sample and Liquid media after samanya and Vishesh shodhan are shown in (**Table1**).

The red hot Mandur Bhasma , when immersed into Triphala Kwath, results into a hissing Sound. Triphala kwatha turned to greenish Black. Finally

a soft and fine, dark Brown powder of specially shodhit was obtained.The color of the bhasma varying from black to brown after ignition. The difference in the color may be due to the number of putas or ignition temperature.

The Prepared Bhasma shows 80%, 84% of total ash content and 20 %, 16% acid insoluble ash content for Mandur bhasma prepared by ignition using Electric Muffle Furnace and by traditional puta system respectively.

The comparative study related to the structure, Morpholgy and elemental content of prepared bhasma is reported below.

3.1 X- ray diffractometer (XRD):

XRD Patterns of Mandur bhasma are shown in (**Fig 6**).

The diffraction pattern of Bhasma at various stages of its preparation is shown in above figure. The presence of sharp diffraction peak shows the highly crystalline nature of bhasma .The sample shows diffraction peak majorly at $2\theta = 35.5^0$, 35.6^0 and 48.3^0in finally prepared bhasma (**Fig 6 a, b**) which indicates the planes 211,211,310 with reference to JCPDS file No. 01-1267 confirming the presence of iron oxide .The synthesized Bhasam also compared with the XRD of marketed sample (**Fig 6c**) which almost shows same crystalline geometry. While sample analyzed after vishesh shodhan (**Fig 6d**) shows the major peak at 48.3^0.

The XRD pattern in final product (Fig 6 a, b)shows that iron oxide is majorly present in the form of Fe_2O_3 and Fe_3O_4(Magnetite) during heating in Bhasmikaran at high temperature (650^0C) Iron oxide formed in its most stable form α-Fe_2O_3. In this Fe_3O_4 is mixture of two states of iron (FeO and Fe_2O_3). FeO is easily converted into its most Suitable form Fe_2O_3.

3.2 Scanning electron microscopy (SEM):

SEM Micrographs of Mandur Bhasma are shown in (**Fig 7**).

The (**Fig 7 a, b, c, d, e, f**) shows surface morphology of sample at various stages of its preparation. The study revealed regular and uniform arrangement of cluster of granules in finally prepared Bhasma which was not observed in raw material. It is clearly observed that the surface area was smooth after shodhan (Purification) treatment. Also study showed the particle size reduction in final product.

3.3 Energy dispersive X- ray analysis (EDAX):

(**Table 2**) shows elemental analysis at various stages of shodhan.

It is observed that percentage of iron in the initial sample decreases after samanya shodhan and Vishesh shodhan treatment. Along with this the various other trace elements are incorporated in the preparation of the bhasma. Bhasma is found to be rich in K, Ca, S, Fe, Na, P. Among all elements Na, K which are responsible for maintaining normal fluid balance are also found to exist in considerable amount [10]. All these elements taken from herbs during purification are act as essential elements to increase the efficiency of drug and seem to be supplement to cure disease. Thus elemental analysis shows that, the nutrient elements present in the Bhasma sample are due to herbal sources used in the purification.

EDX Spectrum of Mandur bhasma is shown in (**Fig 8**).

The above Figure represents the elemental content in prepared Mandur bhasma (**Fig 8 b, c**) . Which was compared with Commercial sample of Bhasma (**Fig 8 d**) and raw material (**Fig 8 a**) used for its preparation. It is observed that along with Fe used in the drug, other elements such as K, Ca, S, Fe, Na, P, and Mg are also present in considerable amount. It is observed that concentration of Iron decreases in final product which is high in raw material.

An EDX spectrum of synthesized bhasma and commercial bhasma shows presence of same elements having nearly same concentration.

3.4 FTIR Analysis of Mandur Bhasma:

FTIR spectrum forMandur bhasma are shown in (**Fig 9**).

FTIR analysis shows the major peaks in all three samples at 1525.74 cm^{-1}, 2332.8 cm^{-1} and 3738.18 cm^{-1} with different intensities.

3.5 BET Analysis of sample:

Area and pore size of Mandur sample prepared by two different methods are mention in above (**Table 3**).

It was observed that pore sizes are 2.5 and 1.9 A^0 for Mandur prepared by using Furnace and

traditional Puta method respectively. The Surface Area was more for sample prepared by traditional method than sample prepared by using furnace it might be due to uneven heating in Puta system.

3.6 NPST Test:

Results of the NPST qualitative Test of synthesized bhasmas are summarized below.

Spotting on 10% Potassium Iodide Paper:

The Observations for the prepared Bhasma are shown in following images,

Phase I (5 minutes): Dark brown central zone, Light brown peripheral segment

Phase II (30 minutes): White central zone, Dark brown middle segment,Light brown peripheral segment(**Fig 10**).

These observations indicated formation of Mandur bhasma and are in agreement with results by Sonkar Niranjan *et al* [11].

Table1: Observations during Samanya and Vishesh Shodhan Process

S. No	Liquid Media	pH	Change in Sample	Change in Liquid Media
	Samanya Shodhan			
1	Sesame oil	6.4	Blackish in color	Blackish and oily
2	Butter Milk	4.5	Blackish Oily	Blackish and oily
3	Cow Urine	7.6	Size reduced	Ammonical smell
4	Sour Gruel/Kanji	3.5	Brittleness increase	Black brown in color
5	Horsegram decoction	6.2	Rate of breaking iron scrap increase	Brownish red decoction turns black
	Vishesh Shodhan			
6	Triphala Decoction	2.3	Iron rust become more brittle	Liquid media turn blackish

Table 2: EDAX analysis of sample during preparation

Elements	C	O	Si	Cl	K	Ca	Fe	S	Zr	Na	P
% WEIGHT											
Initial	10.32	51.42	0.46	-	-	-	37.04	-	-	-	-
Samnya Shodhan	76.81	19.16	-	1.21	1.05	0.28	0.18	0.25	-	0.83	0.23
Vishesh Shodhan	71.54	24.71	-	1.01	0.34	0.40	0.83	0.15	0.56	0.72	-

Table 3: BET analysis of sample

Sample / Parameter	Mandur Prepared by Furnace	Mandur prepared by traditional Puta method
Surface Area	0.932 m^2/gm	3.2 m^2/gm
Pore size	2.5 A^0	1.9 A^0

Fig 1: Chakrikas/Pellets of Bhasma

Fig 2: Ardhagajaput for maran of Mandur

Fig 3: Final Mandur Bhasma

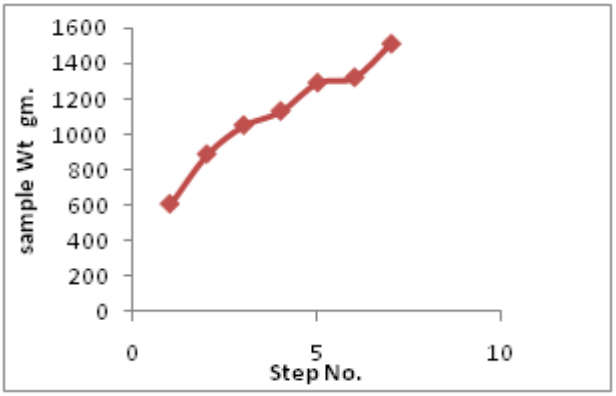

Fig 4: Weight change of sample in Bhanupak Process (Vishesh Shodha

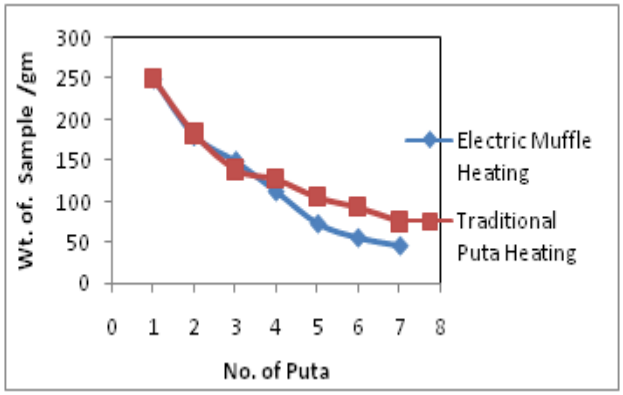

Fig 5: Weight change of sample in maran Process

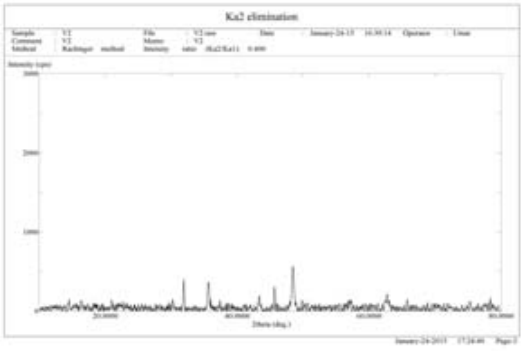

Fig.6a

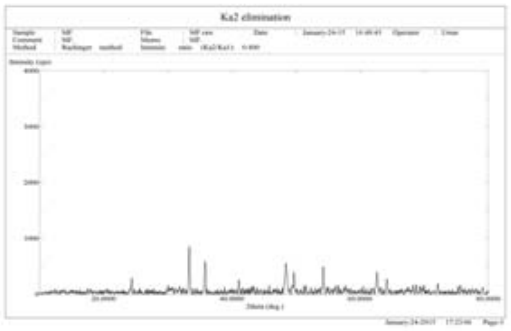

Fig 6b

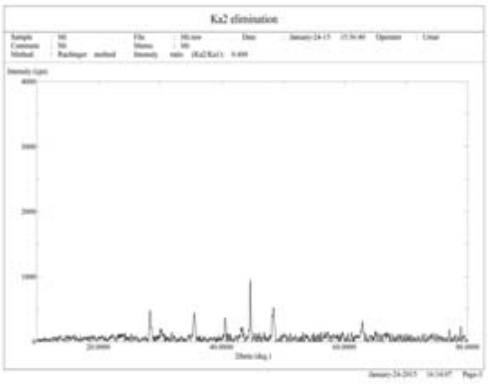

Fig.6c

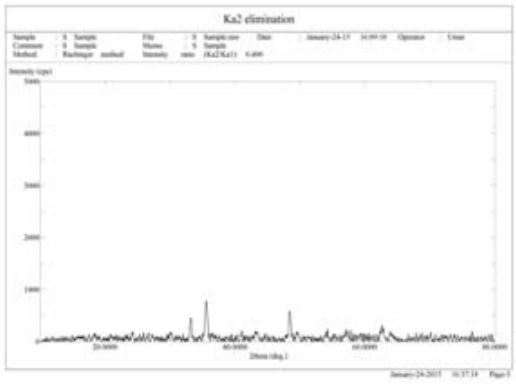

Fig.6d

Fig 6: XRD of Mandur bhasma a) After Vishesh Shodhan b) Prepared using Electric Muffle furnace c) Prepared using traditional Puta System d) Commercial sample

Fig 7a

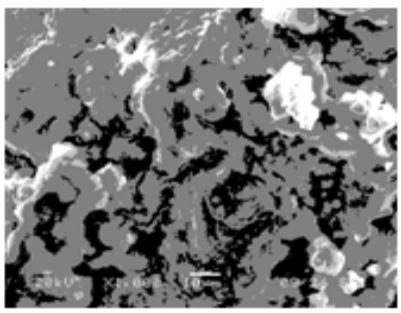

Fig 7b

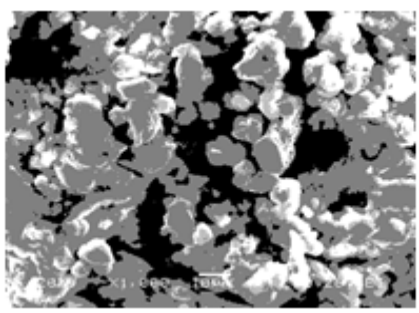

Fig 7c

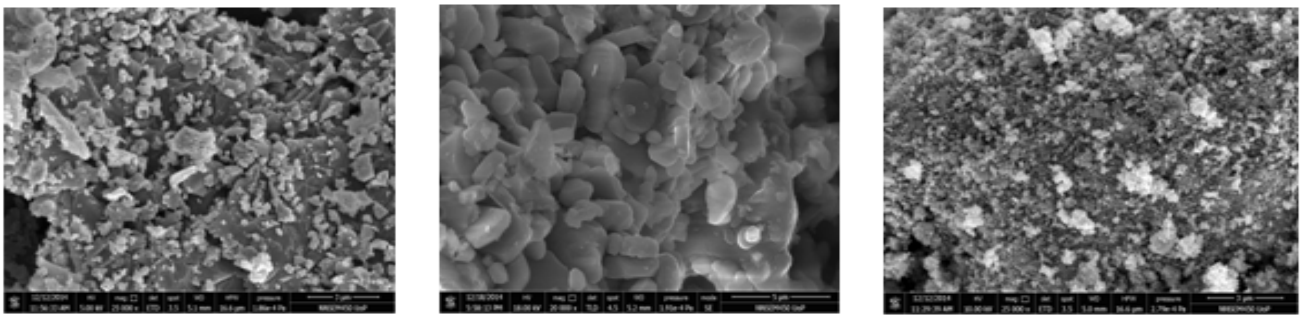

Fig 7d Fig 7e Fig 7f

Fig 7: SEM Micrographs of Mandur bhasma a) Raw b) After samanya Shodhan c)After Vishesh shodhan d) Prepared by Electric Muffle furnace heating e) Prepared by traditional puta heating f) Commercial sample

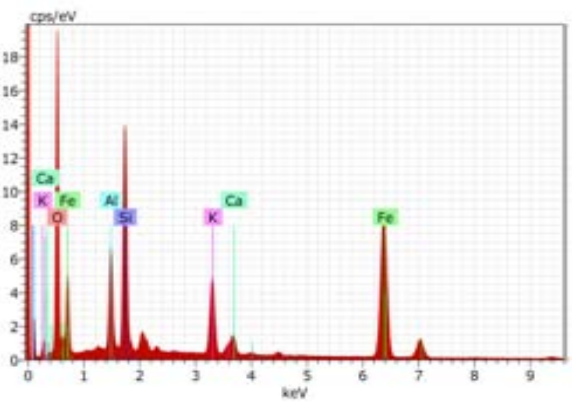

Fig 8a

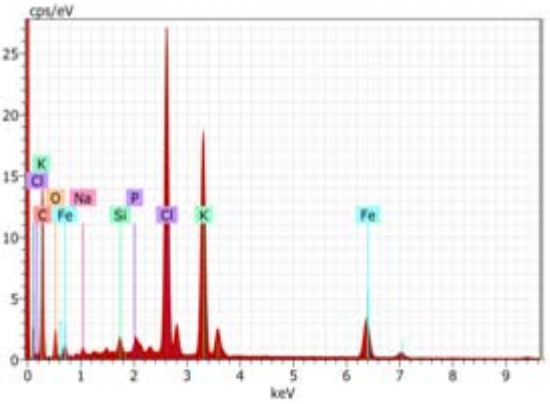

Fig 8b

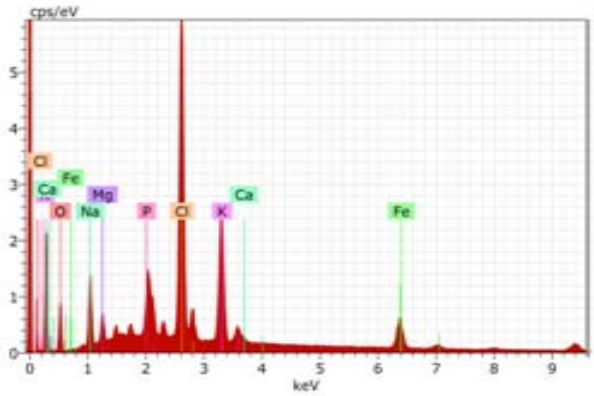

Fig 8c

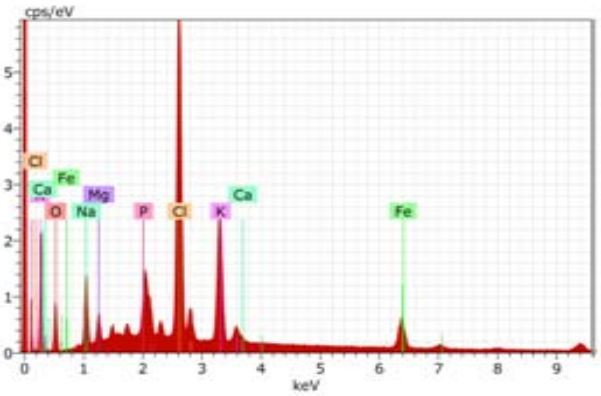

Fig 8d

Fig 8: EDAX Spectrum of Mandur Bhasma a) Raw Material b) Prepared using Electric Muffle Furnace (c) Prepared by Traditional puta system (d) Commercial

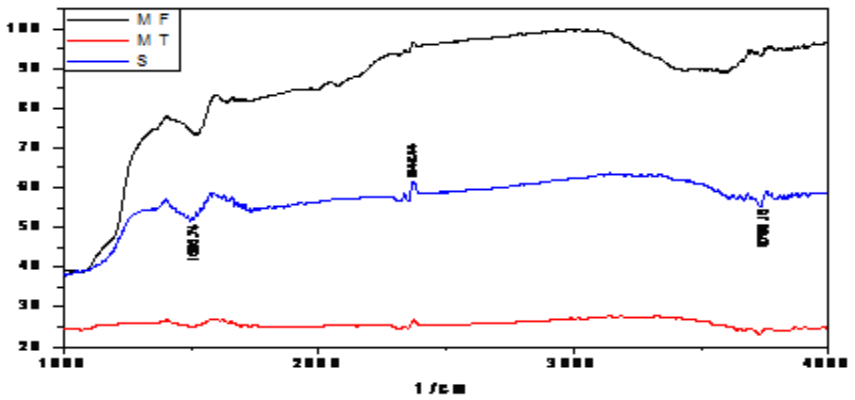

Fig 9: FTIR Analysis of Mandur Bhasma MF: Mandur Prepared by Electric Muffle Furnace heating MT: Mandur prepared by traditional Puta heating S: Marketed sample

Phase 1

Phase II

Fig10: NPST Test for Mandur Bhasma

4.0. CONCLUSION

The XRD pattern in final product shows that iron oxide is mainly present in the form of Fe_2O_3 and Fe_3O_4. It was observed that the iron content in the raw material decreases in final product. The EDAX analysis indicates that elements other than iron are incorporated in the Bhasma during various processes. The SEM image clearly shows the change in morphology and decrease in particle size of the final product. The prepared bhasma satisfied the NPST test of qualitative analysis. It is observed that techniques employed for Purification and calcination of Mandur significantly reduces the Particle size in Mandur Bhasma.

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