Synthesis and Characterization of Pittal Bhasma

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Abstract:

Ayurvedic Bhasmas play very important role in the treatment of various diseases. However, it should be in the pure form to avoid any adverse effects on human health. The present work describes the synthesis of copper and zinc based Pittal bhasma followed by its characterization using modern analytical techniques such as TEM, SEM, EDX, XRD, DLS and FTIR as well as some ayurvedic tests. Synthesis involves traditional method of shodhana, bhavana and marana . For the latter, traditional putas as well as modern electric muffle furnace were also used for heating purpose. The study reveals that Pittal Bhasma prepared by traditional method of heating has 60 % particles in the range of 300-750nm while that prepared by using electric muffle furnace has 65 % particles in 250-750nm range. In both cases the bimodal particle distribution is observed. The study concludes that bhasma can be useful medicine if it is prepared by standard method of preparation and analysis.

Key Words: Pittal Bhasma, Purification, Ayurveda, Traditional Medicine

1. Introduction:

Ayurvedic Bhasmas play very important role in the treatment of various diseases[1]. Bhasmas are unique ayurvedic herbo-metallic preparations useful in various ailments. Starting raw material, various ingredients used during synthesis, trituration process and way of incineration process decides quality of bhasma. The incomplete incineration may result in impurities which can lead to the adverse effects and toxicity. If Bhasma is not prepared properly, it can cause multiple complications such as skin diseases, fever, delusion etc. Hence, Different classical textbooks of Rasashastra have mentioned various parameters for the Standardization of Bhasma[2], as a single method [2-5] is not applicable for determining the accurate formation of a particular bhasma.

The various confirmatory parameters for prepared Bhasma are specified in the various Ayurvedic textbooks and literature[5-9]. Copper and zinc based Pittal Bhasma is used for the treatment of various skin diseases[3,4,9]. We had reported synthesis and characterization of different bhasmas from our laboratory, among which vanga bhasm and Abhrak bhasma were

found to have formation of nanoparticles in it [10,11]. The present work describes the synthesis of Pittal bhasma followed by its characterization using modern techniques such as TEM, SEM, EDX, XRD, DLS and FTIR as well as some ayurvedic tests.

2. Materials and Methods:

Pittal was purchased from the local Market and sheets were prepared in workshop of Chemistry Department, Savitribai Phule Pune University. Pittal Bhasma was synthesized as per the procedure described in ayurvedic literature [6]. It involves Shodhana process (purification of raw material) followed by Bhavana process (Levigation). At the end, Marana process (incineration)was carried out. Incineration was done with traditional putas as well as modern electric muffle furnace (EMF).

2.1 Purification:

Liquefaction and pouring methods were used for purification of raw material. In this process the metals are subjected for purification with various liquid media viz. sesame oil, butter milk, cow's urine, kanji, and decoction of horse gram. The Sheets of Pittal (brass) weighing 99.1 g were used for preparation of Bhasma**.** The metal plates (5×5 cm) were kept on flame and heated till the material becomes red hot.The red hot metal sheets were then quenched in 100 mL of sesame oil taken in earthen pot. Sesame oil was separated, and plates were dried and its weight was recorded. The above procedure was repeated for six more times. Similar purification was carried out using butter milk, cow urine, kanji, horse gram decoction respectively. For each step of purification fresh liquid media was used.

2.1. Special purification (Vishesh shodhana)

In special purification of brass metal, plate processed in previous step was heated till it became red hot and quenched in 100 mL mixture of Vitex nigundo leaves juice and 50 g powder of Curcuma longa Linn (haridra). The process was repeated 3 times. This procedure modifies the properties of the therapeutic material to enhance their potential.

2.**2Levigation:**

The sample after Purification was subjected for levigation. The metal plates were converted into small pieces after purification. The pieces of brass from above step were ground with the leaves of *Calotropis gigantean (Rui)* plant till it was converted into fine powder.

2.3 Incineration:

In this method small pellets were prepared by using paste from levigation step. These pellets were then kept in closed vessel (sharav) and subjected for heating. The sample was slowly heated in electric muffle furnace up to 1100^0C and kept for 3h. The sample was also heated by traditional method. The process was repeated 7 times.

The final form of bhasma was yellowish in colour. It was then analyzed by ayurvedic tests and XRD, FTIR, SEM, EDAX, TEM and DLS techniques

3 Results and Discussion:

3.1 Ayurvedic tests of Pittal Bhasma:

The prepared bhasma was analyzed in view of ayurvedic tests, the results of which are recorded in Table 1

Observations recorded in Table 1 were found to match with those reported in ayurvedic literature.

The major changes observed during purification were that the sample melts after heating, while pouring into liquid media, the sample becomes hard and shiny. At the end of special purification, the sample was converted into small pieces which were more brittle.

The initial weight of raw Pittal was 50 g. The weight of finally obtained Pittal bhasma by traditional method of heating was 25 g and Electric muffle Furnace heating was 30.5 g.

3.1 XRD analysis at different stages of synthesis:

The synthesized 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.The scanning angle was between 10-80 ^{0}C and the rate of scanning was 1^0 /min.

The XRD pattern at each stage of preparation of Pittal Bhasma is shown In Figures 1a and 1b

The presence of sharp diffraction peak shows the highly crystalline nature of bhasma. The XRD spectra of starting material of Pittal Bhasma (Fig.1a) shows the major peaks at $2\theta = 35.5^0$, 35.6^0 48.3^0 and in the sample shows diffraction peak mainly at $2\theta = 42.50^0$, 49.30^0, 49.53^0 and 72.32^0 which indicates the planes 200,202 and 220, which is related to Cu and CuO. Spectra for the starting material after purification(Fig1b) shows similar peaks which are present in staring material except the peak at $2\theta = 72.32^0$.After purification the final product of Pittal Bhasma prepared by incineration with EMF heating and traditional method of heating (Fig.2a and 2b) shows clear phase change and crystalline nature of the sample. The major peaks are observed at $2\theta = 32.68^0$, 39.20^0, 46.18^0 and 56.46^0 in the bhasma prepared by traditional method of heating (Fig.2a) which represents the planes 110,002, 202, 220.While the Pittal Bhasma prepared by electric muffle furnace heating (Fig.2b) shows major peaks at $2\theta = 27.88^0, 29.98^0, 35.32^0, 38.70^0, 48.66^0$, 62.38^0 which is related to planes 110, 002, 202, 200, 220.The peaks corresponds to Cu_2O, Cu as well as Cu_4O_3 with reference to JCPDS file No.

05-0661 and 33-0480.The peaks are observed to be more intense in spectra for Pittal Bhasma incinerated with EMF heating than that of the spectra for the bhasma prepared by incineration with traditional method of heating.

The mean crystallite size calculated by Scherer equation (1) is recorded in Table 2

$$t = \frac{0.9\lambda}{\beta cos\theta} \text{---------- (1)}$$

Where, t =Crystallite size, λ = Wavelength , β = Full width at Half Maxima

An examination Table 2 shows that the mean crystallite size for the initial sample of Pittal Bhasma is 157.48 nm. This decreases after purification to 152.8 nm. Bhasma prepared by incineration with electric muffle furnace heating shows less crystallite size than that of the bhasma prepare by traditional method of heating.

3.2 FTIR analysis during Pittal Bhasma process:

The FTIR spectrum of sample shows peaks in 400- 4000 cm^{-1} region. The FTIR analysis was carried out on SIMADZU-7000. The major peaks obtained from FTIR spectra (Fig.3) for Pittal Bhasma is shown in Table 3.

As can be seen from Table 3 and Fig.3, for the Pittal Bhasma prepared by EMF heating shows major peaks at 519 cm^{-1}, 867 cm^{-1}, 1101 cm^{-1} and 2334 cm^{-1}. But the bhasma prepared by traditional method of heating shows peaks at 481 cm^{-1}, 2134 cm^{-1}, 2327cm^{-1}. The spectra in low frequency region are mainly due to the metal oxide bond such as Cuo, ZnO. The peak intensity is more in case of Pittal Bhasma incinerated with electric muffle furnace heating than the bhasma incinerated with traditional method of heating.

3.3 SEM Analysis during Pittal Bhasma Preparation process:

SEM of Pittal bhasma at various stages of synthesis are shown in Fig.4 and Fig. 5

The SEM analysis was carried out on FEI Nova-nano SEM-450. The scanning electron micrograms at various stages of preparation are shown in Figs.4and Fig.5

An examination of Fig. 4a –4b reveals regular and uniform arrangement of cluster of granules in finally prepared bhasma, which was not observed in raw material (Fig.4a). It is clearly observed that the surface area was smooth after special purification (shodhan) treatment (Fig.4b) and organic constituents are adsorbed on the surface of the sample during preparation. The particle size observed after special purification is 250nm - 1μm. Hence, it is

clear that purification process reduces particle size. The bhasma prepared by traditional method of heating shows the uneven size of the bhasma particles (Fig.5a). The size of Pittal Bhasma prepared by EMF heating is less as compared to the bhasma prepared by incineration with traditional method of heating. This may be due to the uneven heating of sample in traditional method. .The size of particles in Pittal Bhasma prepared using electric muffle furnace is between 50-100nm (Fig5b), while that of the bhasma prepared by traditional method of heating shows clusters of particles within 250nm -1μm (Fig.5a). It also shows some rod like structure.

3.5 EDX Analysis during Pittal Bhasma process:

Fig.6 to Fig. 9 show EDX spectra of Pital Bhasma at different stages of preparation (using of Bruker XSHLASH-6 I30 electron microscope) and Table 4 to Table 7 includes elemental content obtained from EDX analysis of Pittal Bhasma at various stages of preparation

The EDX spectra of starting material of Pittal Bhasma (Fig.6 and Table 4) shows the Cu and Zn in major amount along with some trace elements such as S, Fe, C. After purification (Fig.7 and Table 5) it was observed that other elements are incorporated in bhasma Cu, O, S, C. The final product of Pittal Bhasma prepared by incineration with traditional method of heating (Fig.8 and Table 6) shows O, Si, Fe, Pb, Ca, Mg and P along with the Cu ad Zn. The concentration of Cu is observed to be decreased , while the bhasma prepared by EMF heating(Fig.9 and Table 7) shows the presence of Cu, Zn, Al, Si, Mg, P.C,O and K. The source of elements other than Cu and Zn may be medicinal plants used during preparation of Pittal Bhasma. All these elements are obtained from the herb and useful to increase the efficacy of the bhasma. These are seems to be additional supplement for curing the disease. Thus, the elemental analysis shows the nutrient elements present in the bhasma sample are due to the herbal material used in the preparation.

3.6 TEM analysis during Pittal Bhasma

TEM analysis of Pittal bhasma at various magnifications (using TecnaiG2U-twin200Kv Lab6FEI Netherlands) are shown in Fig. 10

The bright spots in SAED pattern support the highly crystalline nature of the Pittal Bhasma. The TEM image Further shows spongy nature in bhasma. The inter planer distance in Pittal Bhasma prepared by incineration with EMF heating (Fig.10b) is 0.268 nm while that of the

bhasma prepared by traditional method of heating (Fig.10 a) is 0.299 nm. Thus, TEM analysis also supports that the bhasma prepared by EMF heating is better method than traditional method of heating.

3.7 DLS analysis During Pittal Bhasma preparation process:

The diffusion coefficient, effective diameter and polydispersivity of Pittal Bhasma are reported in Table.8

As can be seen from Table 8, the dynamic light scattering study of bhasma shows that the effective diameter for the bhasma prepared by EMF heating is less as compared to the bhasma prepared by traditional method of heating.

Conclusions:

XRD of Pittal Bhasma shows the presence of CuO and granular appearance and polycrystalline nature. ·EDAX analysis of Pittal bhasma shows incorporation of number of nutrient elements.

SEM of Pittal Bhasma Shows particles with change in morphology. The Bhasma prepared by EMF heating are smaller in size than the Traditional Method of heating. FTIR for traditional method shows major peaks for C-H ,C=C ,Cu-O bond. Pittal Bhasma prepared by traditional method of heating has 60 % particles in the range of 300-750 nm while that prepared by using electric muffle furnace has 65 % particles in the range of 250-750 nm. In both cases the bimodal particle distribution is observed.

Acknwoledgement:

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Figures:

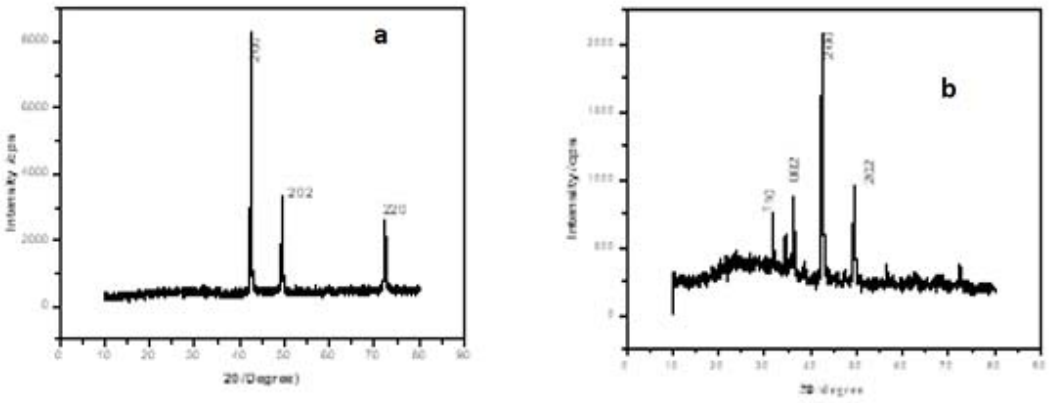

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Fig. 1 XRD spectra of Pittal Bhasma a) before purification b) after purification

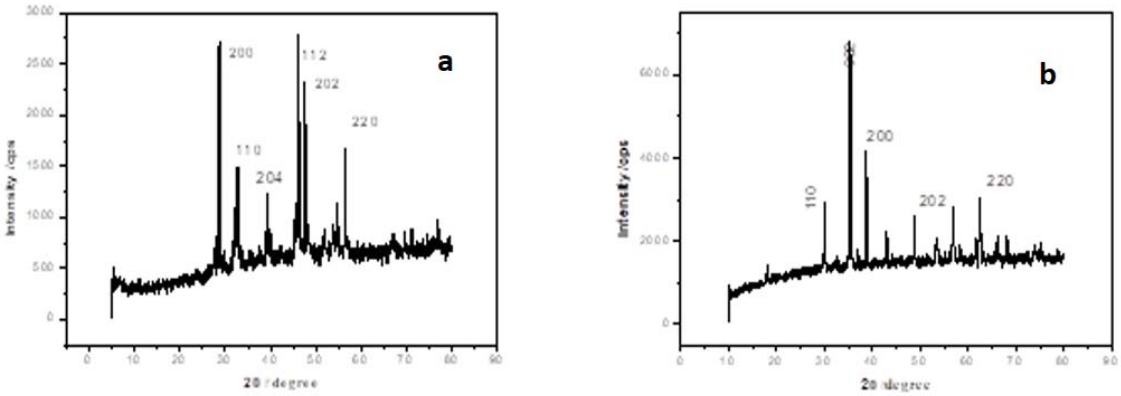

Fig. 2 XRD spectra of Pittal Bhasma prepared by a) Traditional method of heating b) EMF **heating**

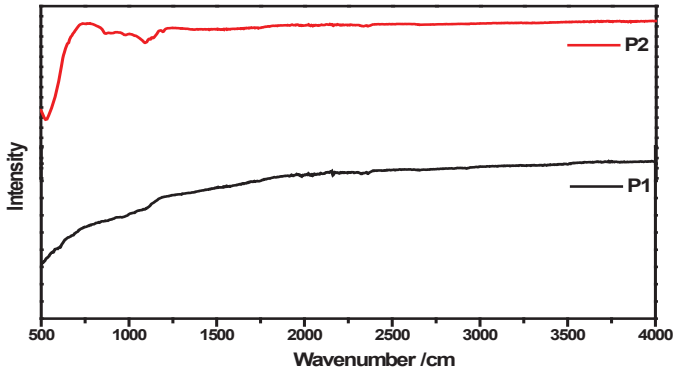

Fig.3 FTIR spectra of Pitta Bhasma

P1: Pittal Bhasma prepared by traditional method of heating
P2 :Pittal Bhasma prepared by EMF heating

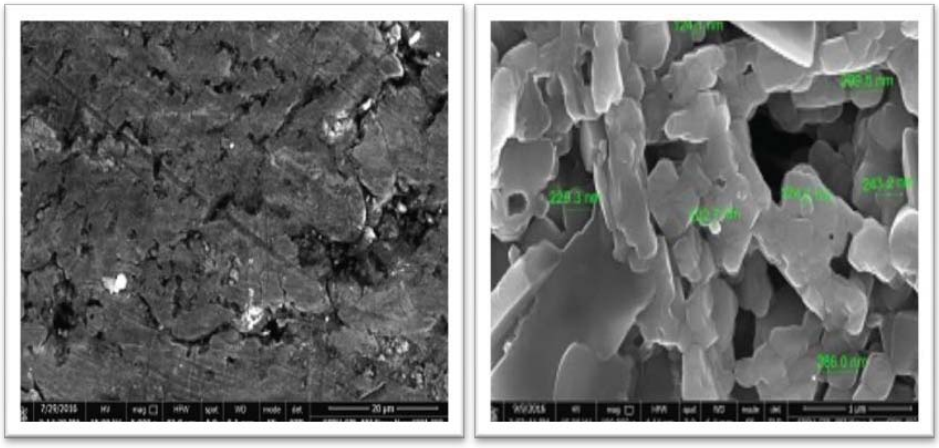

Fig. 4 SEM micrograph of a) starting material b) after Purification of Pittal Bhasma

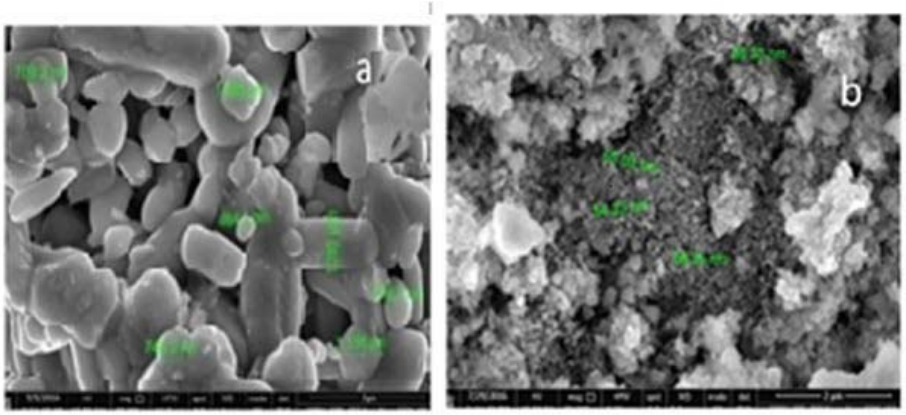

Fig.5 SEM Micrograph of Pittal Bhasma a) prepared by traditional method of heating b) prepared by EMF heating

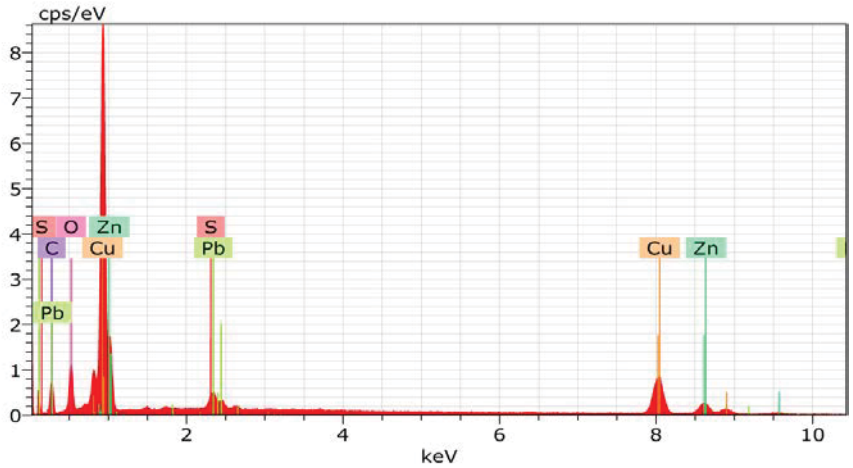

Fig.6 EDX spectra of starting material of Pittal Bhasma

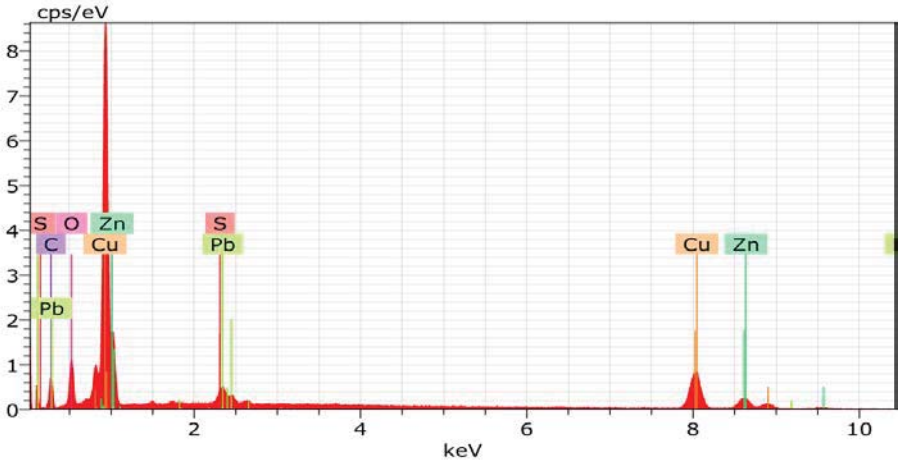

Fig. 7 EDX spectra of Pittal Bhasma after purification

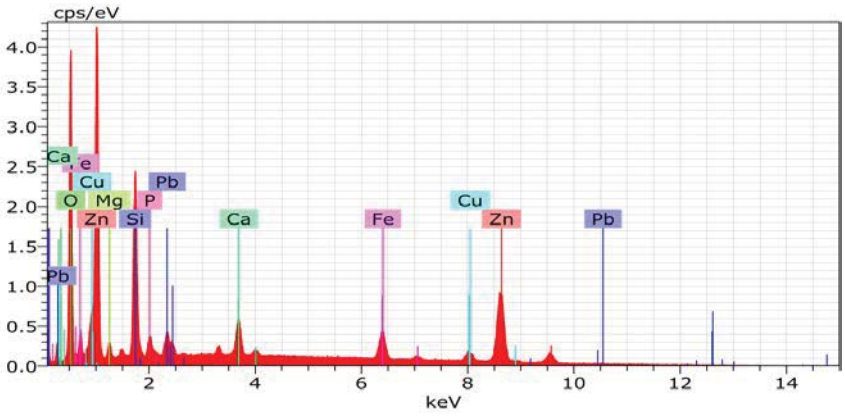

Fig.8 EDX spectra of Pittal Bhasma prepared by traditional method of heating

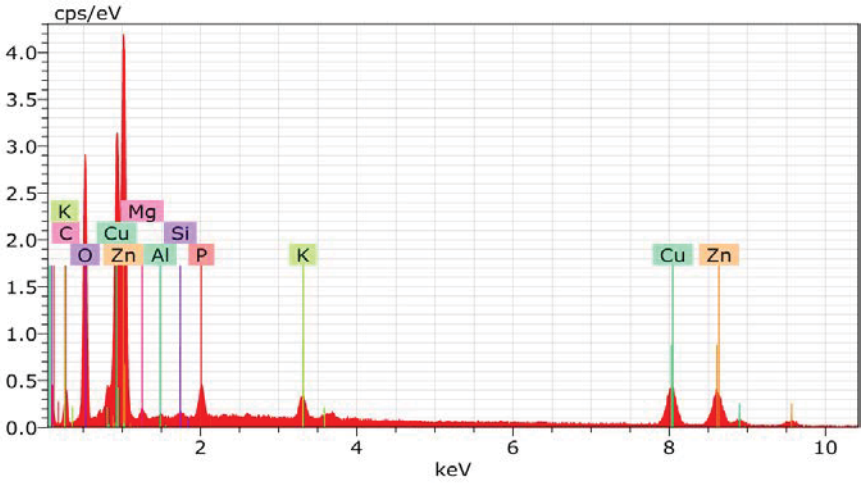

Fig.9 EDX spectra of Pittal Bhasma prepared by EMF heating

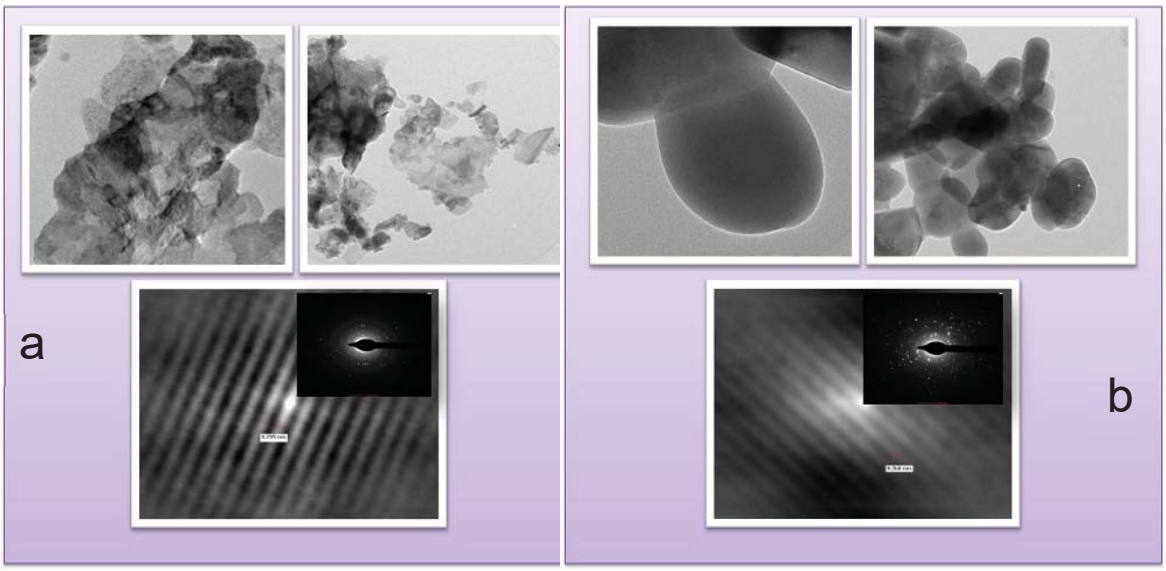

Fig. 10 TEM, HRTEM, SAED of Pittal Bhasma prepared by a) traditional method of heating b) EMF heating

Tables:

Table 1 Ayurvedic tests of Pittal Bhasma

TEST	P1	P2
Odor	Odorless	Odorless
Colour	Dark Brown	Dark brown
Nishchandratvam	+ve	+ve
Rekhpurnatvam	+ve	+ve
Varitratvam	+ve	+ve
Unnam	+ve	+ve

P1: Pittal Bhasma prepared by traditional method of heating
P2:Pittal Bhasma prepared by EMF heating

Table 2 Crystallite size during preparation of Pittal Bhasma

PittalBhasma	2θ (degree)	d value	Mean Crystallite size /nm
P1	42.50,49.30, 49.53,72.32	2.12,1.83,1.83, 1.30	157.48
P2	42.50,49.38	2.12, 1.84	152.80
P3	32.68,39.20,46.18,56.46	2.73,2.29, 1.96, 1.62	139.70
P4	27.88,29.98,35.32,38.70, 48.66,62.38	3.19,2.97,2.53,2.32, 1.86, 1.42	126.39

P1: Starting material of Pittal Bhasma
P2: Starting material of Pittal Bhasma after purification
P3: Pittal Bhasma Prepared by traditional method of heating
P4: Pittal Bhasma Prepared by EMF heating

Table: 3 FTIR analysis of Pittal Bhasma

Functional group	Absorption wavenumber /cm^{-1}	
	P1	P2
Cu-O,Zn-O	431,461	519
C-O	-----	1101
O-H	2334	2334

P1: Pittal Bhasma prepared by traditional method of heating
P2 :Pittal Bhasma prepared by EMF heating

Table: 4 EDX analysis starting material of Pittal Bhasma

Element	Conc./%	Element	Conc./%
Zn	15.90	K	0.10
Cu	72.61	Fe	1.92
C	3.16	Mg	0.01
Na	0.52	S	16.02

Table 5 EDX analysis of Pittal Bhasma after purification

Element	Conc./%	Element	Conc./%
Zn	22.43	S	0.72
Cu	55.62	C	6.69
O	8.01	Pb	4.09

Table 6 EDX analysis of Pittal Bhasma prepared by traditional method of heating

Element	Conc./%	Element	Conc./%
Zn	15.17	Pb	0.50
Cu	35.17	Ca	2.69
O	15.01	Mg	1.33
Si	5.69	P	0.23
Fe	3.17		

Table 7 EDX spectra of Pittal Bhasma prepared by EMF heating

Element	Conc./%	Element	Conc./%
Zn	26.04	K	2.10
Cu	29.62	P	1.92
O	20.23	Mg	0.82
C	6.44	Si	0.23
Al	0.18		

Table 8 DLS analysis during Pittal Bhasma

Sr. No.	Sample No.	Diffussion coefficient /10 $^{-9}$cm^{2}S $^{-1}$	Effective diameter /nm	Polydispersivity
1	P1	8.02	611.21	0.279
2	P2	8.09	551.04	0.267

P1: Pittal Bhasma prepared by traditional method of heating
P2: Pittal Bhasma prepared by EMF heating

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