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Research Article

Chemical Characterization of *Lauha Bhasma* by X-Ray Diffraction and Vibrating Sample Magnetometry

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Abstract

Herbomineral formulations are unique to Ayurveda. *Lauha bhasma* is an Ayurvedic metallic preparation used in iron deficiency anemia. Our emphasis in the present paper is on chemical characterization of *Lauha bhasma* at different steps by following X-ray Diffraction (XRD) study and Vibrating Sample Magnetometry (VSM) study. XRD study was conducted for the samples after *Sthalipaka*, 1st *Puta*, 5th *Puta*, 10th *Puta*, 15th *Puta* and 20th *Puta* and VSM study was conducted for samples after 5th 10th and 20th *Puta*. Fe_3O_4 and γ-Fe_2O_3 may be accepted as chemical characterization of *Lauha bhasma* in broader way as it has been explored in this project by applying more sophisticated analytical technique like XRD and VSM.

Key words: Herbomineral formulations, *Lauha bhasma*, XRD, VSM, γ-Fe_2O_3, Fe_3O_4

Introduction

Ayurvedic Herbomineral formulations are unique in terms of their minimal dose, quick action, palatibility and wider therapeutic applicability. The metals and minerals are not used as such. They are subjected to some needful procedures called "pharmaceutical processes" which include *shodhana*, *marana* etc. which convert the raw drug into a suitable compound form.

Earlier, the quality of the herbomineral preparation was subjected to review, but the methods were very crude like for test of completion of finished *bhasma* procedures like *rekhapurnata, varitara, nischandratva* etc were performed. With technological development the patients or the physicians seek assurance for the quality, safety and efficacy of any medicine, especially for export in the western countries. Therefore, quality control for herbal preparations and *bhasmas* is essential as many of them contain chemical entity as Cu, Zn, Fe, As, Hg and Pb. Current issue of quality control method of identification of compounds made up of various metals/ mineral requires chemical characterizations by XRD. At the same time to reveal the magnetic property of iron it is also required the *Lauha bhasma* to screen under VSM. Hence, in present paper observations of *Lauha bhasma* found in XRD & VSM are mentioned.

Lauha bhasma an Ayurvedic preparation is one of the best medicines for iron deficiency anemia. It is better absorbed from GIT and is devoid of the side effects. *Lauha bhasma* is prepared by following steps of *Samanya shodhana,*

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Vishesha shodhana, Bhanupaka, Sthalipaka and *Putapaka. Putapaka* was conducted at a temperature of 550^0C-600^0C in electric muffle furnace. It took twenty *puta* (shown in Figure 01, 02, and 03) for complete conversion of material into finally prepared *bhasma* form (therapeutic form of material). The finally prepared *bhasma* was identified on physicochemical parameters. The physical parameters were colour (blackish red-*pakwajambu phala varna*), *rekhapurnatva* (on rubbing the *bhasma* between index finger and thumb the minute particles entered into the furrows of finger), taste (the *bhasma* was found to be tasteless) and *varitara* (The *bhasma* was found approximately 75% *varitar*). The chemical parameter was *apunarbhava* (No lustrous particles seen which was indicative of absence of free metal in the *bhasma*).

Our emphasis in the present paper is on chemical characterization of *Lauha bhasma* at different steps by following X-ray Diffraction (XRD) study and Vibrating Sample Magnetometry (VSM) study. To find the major phases present at different levels of pharmaceutical procedures is the main aim of conducting these tests.

In the process of *samanya sodhana* the iron turnings were heated till red hot and quenched (*nirvapa*) in different medias for seven times i.e. *tila taila*, *takra*, *gomutra*, *kānji* and *kulattha kwātha* simultaneously.(Ratna Samuchchya, 5/13)(1) For *visesa sodhana* the process of *nirvapa* in *triphala kwatha* was adopted and repeated for seven times. (Ratna Samuchchya, 5/102)(2) The process of *marana* was completed in three steps viz. *bhanupaka*, *sthalipaka* and *putapaka*. (Rasa Tarangini, 20/ 24, 28, 52)(3) In the process of *bhanupaka* the *lauha churna* procured after *visesa sodhana* was kept in sunlight along with *triphala kwatha* till complete drying. The process was repeated for seven times. *Sthalipaka* and *Putapaka* are specific heating methods. In *Sthalipaka* the material (iron turnings) was boiled along with *triphala kwatha* till drying and in the process of *Puta* the material was triturated with *triphala kwatha* and subjected to heating (at a temp. 550^0C- 600^0C) in electric muffle furnace.

XRD study was conducted for the samples after *Sthalipaka*, 1st *Puta*, 5th *Puta*, 10th *Puta*, 15th *Puta* and 20th *Puta* and VSM study was conducted for samples after 5th, 10th and 20th *Puta*.

Materials & Methods

X- Ray Diffraction:

Principle: X-ray powder diffraction is most widely used non-destructive technique for investigation of structural properties of crystalline materials. X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample.

Sample Preparation and Experimental: 200mg of sample was taken. Sample was grinded to fine powder. Powder less than ~10 μm (or 200-mesh) in size is preferred. The powder was mounted on sample holder and subjected for reading.

Vibrating Sample Magnetometer

Principle: A vibrating sample magnetometer (VSM) operates on Faraday's Law of Induction, which tells us that a changing magnetic field will produce an electric field. This electric field can be measured and can tell us information about the changing magnetic field. A VSM is used to measure the magnetic behavior of magnetic materials.

Experimental: The magnetic measurements (M vs H) of the powder samples (after 5, 10 and 20 Putas) were performed using a Vibrating sample magnetometer (VSM, Lakeshore, Model 7410) at 300 K. The principle of VSM is the measurement of the electromotive force induced by magnetic sample when it is vibrated at a constant frequency in the presence of a static and uniform magnetic

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field. The VSM is operated up to 2 T at a vibration frequency of 82 Hz. The magnetic moment was calibrated with standard sample of Ni which had known saturation magnetization (M_S of 6.92 emu at 0.5 T). A small amount of powder was weighed and tightly held in the sample holder to avoid movements inside the sample holder. The amount of sample was 17.6, 18.3 and 15.4 mg for sample no. 7, 8 and 10 (Fifth, Tenth and Twentieth Puta) respectively.(4)

Observations & Result:

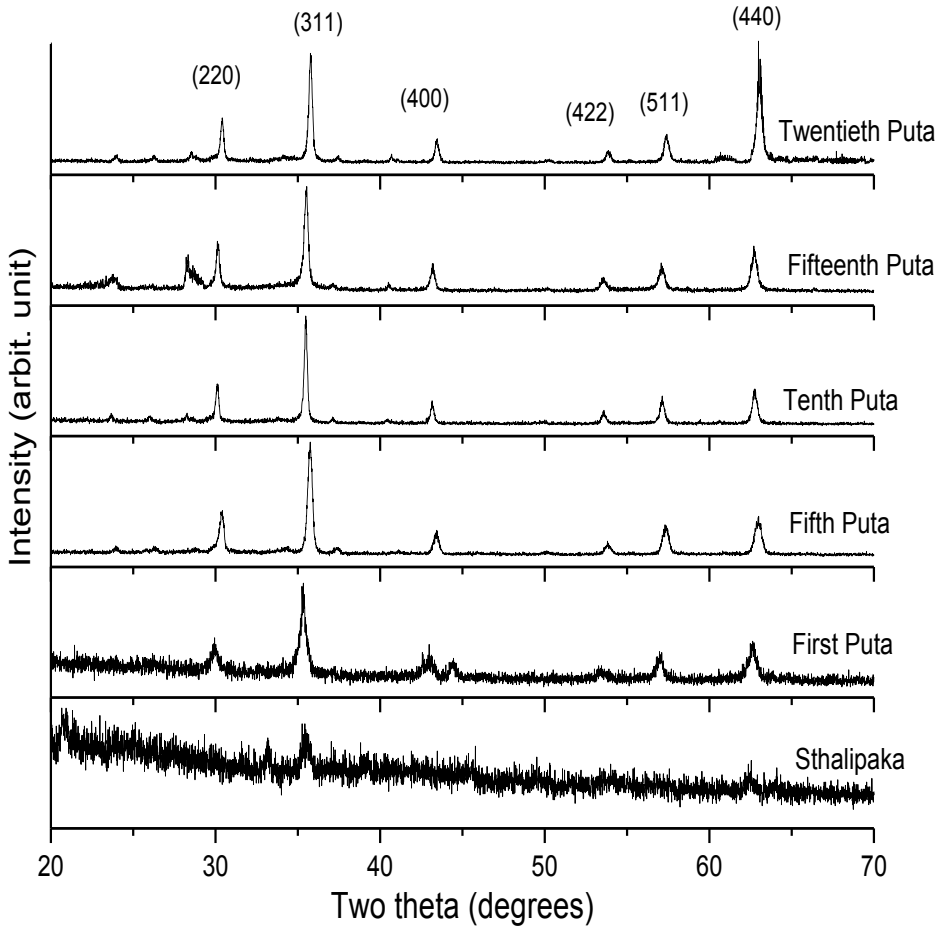

Fig1.1 : XRD pattern of all the samples

Fig.1.1: X-Ray diffraction of all the samples was done by Rigaku DMAX powder X-Ray diffractometer with Cu-Kα radiation. XRD pattern is shown in Fig. 1 and the standard data was taken from ICDD-4+2007 version. Pattern indicates that crystallinity in the material increases successively from *Sthalipaka* to *Twentieth Puta* because the intensity of the peaks is increasing continuously. The patterns reveal that the diffraction peaks corresponding to (220), (311), (400), (422), (511) and (440) planes of γ-Fe_2O_3 and Fe_3O_4 are present (ICDD PDF Card No. 00-001-1111 of Fe_3O_4 and Card No. 00-002-1047 of γ-Fe_2O_3 Annexure 1).

Since the peak position for both the phases i.e. Fe_3O_4 and γ-Fe_2O_3 appears at approximately same two theta values, normal XRD can not distinguish between these two phases. In the entire pattern except the pattern corresponding to Twentieth *Puta,* the (311) plane peak is most intense peak which means that Fe_3O_4 phase is dominating phase. From the pattern it is seen that the intensity of the peak corresponding to (440) plane increases continuously from *Sthalipaka* to Twentieth *Puta* which may be due to the increasing proportion of γ-Fe_2O_3 in prepared sample because the intensity of (440) plane peak is most intense peak in the diffraction pattern of γ-Fe_2O_3 (PDF Card No. 00-002-1047). In the sample Twentieth *Puta* the intensity of the peaks corresponding to (440) plane and (311) plane is comparable to each other which indicates that Fe_3O_4 and γ-Fe_2O_3 may be present in approximately equal proportion.

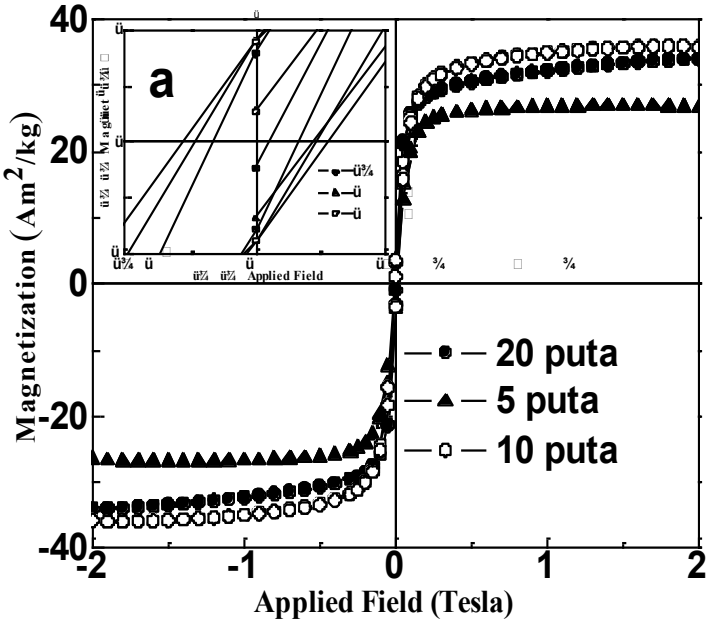

Fig 1.2. Hysteresis loops measured in VSM for different samples

Fig 1.2: Magnetic measurements suggest that all the three samples are magnetic in nature. There magnetization value was found to be 27, 36 and 34 Am^2/kg at 2 T external field for the samples which were undergone 5^{th} , 10^{th} and 20^{th} *Putas* respectively. This suggests the samples are either magnetite (Fe_3O_4) or maghemite (γ-Fe_2O_3). However, the lower value of the magnetization for these samples compared to the aforementioned magnetic iron oxides is because of the presence of the organic materials associated with the samples whose weight were also included while during the measurement. The increase in the magnetization value at higher *Puta* could be attributed to the decrease of organic materials which was added during intermediary pharmaceutical processes. The inset of the figure indicates that all three samples displayed hysteresis loop which suggest that these *Lauha bhasmas* are multi-domain.

Discussion:

XRD pattern is revealing that the crystallinity of material is increasing successively from *Sthālipāka* to 20^{th} *Puta*. In the sample after *Sthālipāka* no intense peak was observed which is revealing amorphous nature of material. At the level of *Sthālipāka* there is not complete oxidation of *Lauha* and due to incorporation of *triphalā kwātha* residues

the material has got changed to amorphous mass.

In every sample, after further processing i.e. subjecting to *Puta* the peaks get intensified which are suggesting that iron is getting converted to a compound form that is iron oxide and thus converting the material to crystalline form which is showing intense peak.

In all the samples except 20th *Puta*, the (311) plane peak is most intense peak which means that Fe_3O_4 is dominating phase. It is seen that peak corresponding to (440) plane increase continuously from *Sthālipāka* to 20th *Puta* which may be due to the increasing proportion of γ-Fe_2O_3.

The above finding suggests that during further process of *Puta* the material gets oxidized and in the beginning the major phase is Fe_3O_4 but up to stage of finally prepared *bhasma* γ-Fe_2O_3 and Fe_3O_4 are in equal proportion.

The Vibrating Sample Magnetometry (VSM) study is also supporting the above finding of XRD. The VSM study was carried out of three samples i.e. after 5th *Puta*, 10th *Puta* and 20th *Puta*.

The study suggests that all the three samples are magnetic in nature. The magnetization value was found 27, 36 and 34 Am^2/Kg at 2T external field it means that the compound is either Fe_3O_4 (Magnetite) or γ-Fe_2O_3 (Maghemite).

Conclusion:

Fe_3O_4 and γ-Fe_2O_3 may be accepted as chemical characterization of *Lauha bhasma* in broader way as it has been explored in this project by applying more sophisticated analytical technique like XRD and VSM.

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Abbreviations:

XRD	:	X- Ray Diffraction
VSM	:	Vibrating Sample Magnetometry
T	:	Tesla
ICDD	:	International Centre for Diffraction Data

ANNEXURE

Table No.1.1: showing 2θ value and d-spacing of different samples and standard values from ICDD.

S.No.7 (5th Puta)		S.No.8 (10th Puta)		PDF Card No.00-001-1111 (Fe_3O_4)		PDF Card No.00-002-1047 (γFe_2O_3)	
2Θ	**d space**	**2Θ**	**d space**	**2Θ**	**d space**	**2Θ**	**d space**
		23.67	3.756				
		25.97	3.428				
		28.22	3.16			35.7431	2.5100
30.36	2.942	30.09	2.967	30.0634	2.9700	30.4837	2.9300
35.72	2.512	35.4671	2.529	35.4512	2.5300	33.7963	2.6500
37.36	2.405	37.10	2.421				
43.38	2.084	43.13	2.096	43.0368	2.1000	43.6923	2.0700
53.83	1.702	53.57	1.709	53.5459	1.7100	54.2312	1.6900
57.38	1.605	57.11	1.611	57.1665	1.6100	57.557	1.6000
62.88	1.477	62.73	1.48	62.7262	1.4800	62.7262	1.4800

Table No.1.2: showing two theta value and d spacing of different samples

S.No.5 (Sthalipaka)		S.No.6 (1st Puta)		S.No.9 (15th Puta)		S.No.10 (20th Puta)	
2Θ	**d space**	**2Θ**	**d space**	**2Θ**	**d space**	**2Θ**	**d space**
				23.77	3.74	23.98	3.708
				28.23	3.159	26.22	3.396
		29.94	2.982	28.65	3.113	28.53	3.126
30.01	2.975			30.12	2.965	30.38	2.94
35.48	2.528	35.27	2.543	35.491	2.527	35.7521	2.509
		42.59	2.121			37.39	2.403
		43.06	2.099	40.50	2.225	40.66	2.217
		43.14	2.095	43.17	2.094	43.43	2.082
		44.37	2.04			44.79	2.022
				53.48	1.712	53.83	1.702
		56.98	1.615	57.08	1.612	57.35	1.605
62.48	1.485	62.62	1.482	62.73	1.48	62.9979	1.474

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Figure 01: Raw material (iron turnings)

Figure 02: Pellets after 4th Puta

Figure 03: Finally prepared bhasma (after 20th Puta)