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CHARACTERIZATION OF LAUHA BHASMA

Research Article

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Abstract

The research methodologies of ancient and modern medicinal science are different but objectives behind them remain the same. Hence in modern day combined practice of Ayurveda, the Indian traditional medicine, testing parameters using traditional and modern analytical methods is required for understanding the Ayurveda in most scientific manner. Iron is an important element for the body system in ancient ayurveda the selection of best iron source and the processing technology to prepared lauha bhasma is more appropriate but there is a need to explain the changes taken place during process of bhasma by using most shofisticted analytical technology hence in present study the analysis of lauh bhasma was done by performing Loss on drying, Determination of Ash value, Determination of Acid insoluble, SEM and, EDAX Analysis,FTIR Analysis and there results has been incorporated in present study. herefore, the present study is taken up to prepare *Lauha Bhasma by using different raw materials i.e. tikshna and kanta lauha. a*nd characterization of lauha bhsma by using traditional parameters as well as modern techniques and there result has been included in present study. The major finding of present study is Scanning electron microscopy along with EDX and FTIR studies proved the conversion of the two starting material viz. iron turnings and magnetite ore (Fe3O4) into a mixture of α-Fe2O3 and γ- Fe2O3 along with oxides of some trace elements.

Key Words: *Lauha, Teekshna Lauha, Kanta Lauha, Lauha Bhasma, Puta*

Introduction

In the medical field it becomes mandatory to study complete analytical profile of a drug for a better understanding of its efficacy. Hence a step is made through this study to provide analytical details of lauha bhasma prepared by using different type of raw materials i.e *tikshna and kanta lauha.* Among the group of *rasaushadhi* It is well known that iron is important in a human body for the formation of haemoglobin, myoglobin, and other substances such as cytocromes, cytochrome oxidase, peroxidase and catalyse. Ayurveda prescribes a detailed protocol (1) for the preparation of lauha bhasma by using different raw materials. These are (i) *Teekshna Lauha* (turnings of pure iron) (ii) *Kanta Lauha* (A magnetic form of iron ore source viz. magnetite (Fe3O4) mineral). There were a few attempts of systematic study of preparation of *Lauha Bhasma* starting from mild steel turnings in the recent past (2,3).

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Aim

In present study is to undertake a detailed characterization of the drug prepared by the prescribed protocol from the above mentioned standard raw materials with a motivation to identify the best raw material for the production of *Lauha Bhasma*. There are various kinds of parameters adopted in this regard. In this section some of the studies conducted over *Lauha bhasma* have been mentioned.

Materials and methods

The pharmaceutical study was conducted in three batches which are mentioned as below.

Batch "A" : *Teekshna lauha*
Batch "B" : *Kanta lauha*
Batch "C" : *Kanta lauha*

Batches B and C differ in the regions from which sampling was done from the mineral source.

Materials

Armco grade iron i.e. a nearly pure iron with very low carbon content was taken in the form of a rod and turnings from it were collected while machining on a lathe and this process done in metallurgy department BHU. Magnetite iron ore was sourced from Mayurbhanj district iron ore mines through National Metallurgical Laboratory, Jamshedpur, India. *Tila taila* was collected from Ayurvedic Pharmacy. *Gomutra* was collected from Dairy farm, BHU and Triphala and Kulattha collected from local herbal market, Varanasi.

Pharmaceutical Procedure

Lauha bhasma was prepared in three batches i.e. Batch-A, Batch-B and Batch-C in which Batch-A is prepared from *Teekshna lauha* whereas Batch B and C were prepared from *Kanta lauha*. In the literature of *Rasa shastra* various methods of *sodhana* and *marana* for *lauha* are mentioned which render the metal and mineral into the form of bhasma.

Sodhana process was carried out in two steps. First is *samanya sodhana* in which red hot iron was dipped in *taila, takra, gomutra, kanji, kulattha kwatha* respectively for seven times in each media. For *visesha sodhana triphala kwatha* was used. After *sodhana*, whole material changed into black colour and big particles got converted into smaller size with increased brittleness in all three batches (1).

After *sodhana, bhanupaka* and *sthalipaka* processes were carried out. In *bhanupaka, triphala kwatha* was mixed with *lauha churna* and whole material was kept in sun light till complete drying took place. This process was repeated for a total of seven times. Then *sthalipaka* was carried out in which *lauha churna* procured from *bhanupaka* was taken in an iron container and *triphala kwatha* was added in sufficient quantity. Then it was kept over a heating device and given intense heat till all the water content got completely evaporated. This process was repeated for six more times (total seven times).

For the *puta* process, instead of adopting a traditional method described in Ayaurvedic texts(2), a modified electric muffle furnace was used to ensure controlled and regulated heating and to standardize the conditions. All batches were prepared at the same temperature i.e. 6000C. Batch A was prepared in 20 *puta*s, whereas Batch B and C were both prepared in 18 *puta*s. For the preparation of *bhasma, triphala kwatha* was used as intermediary substance for *bhavana*.

Batch "A" was maintained at the temperature of 6000C for one hour. After first *puta* colour of pellets was black and surface of pellets was rough. After *puta* whole material in the container was reduced to half of its original volume, because the *triphala* contents which were added during *bhanupaka* and *sthalipaka,* burnt off during *putapaka*. After second *puta* colour of pellets was bluish black.

But surface of some pellets were brown in colour which after trituration again turned black in colour. After fifth *puta* pellets were found to be soft in consistency and were easily breakable. Surface was also found to be smooth. During sixth *puta* pellets turned reddish brown in colour. After 10th *puta* hardness of pellets increased and colour of pellets turned to reddish black besides appearance of black spots over some pellets. After 15th *puta* 50% *bhasma* was positive in *rekhapurna* and 40%*bhasma* was found positive in *varitara* test. After 20th *puta* colour of *bhasma* was blackish red i.e. *pakvajambuphala varna* and *bhasma* was 95% *rekhapurna* and 75% *bhasma* was found positive in *varitara* test and these two tests are done by doing by weighing the materials.

Batch "B" was prepared in the same way and at the same temperature which was adapted for batch "A". During 1st *puta* it was very difficult to make pellets due to coarseness of the powder and after *puta* colour of pellets was black and surface of pellets was rough. After 3rd *puta* colour of pellets was reddish brown on the exterior surface but interior surface was black in colour. There were cracks on the external surface of pellets. After trituration it turned to black colour again. After 5th *puta* pellets were soft in consistency and easily breakable. Colour of pellets was reddish brown and surface of pellets was slightly rough, as well as 25% *bhasma* was *rekhapurna*. After 10th *puta* pellets were little hard when compared to previous *puta*s and colour of pellets also turned reddish black. Surface of pellets was smooth. After 15th *puta* pellets were slightly hard. Blackish spots were found on the surface of the pellets. *Bhasma* became 60% *rekhapurna* and 50% *bhasma* was found positive in *varitara* test. After 18th *puta* pellets became now soft and easily breakable by hand. Colour of *bhasma* was blackish red i.e. *pakvajambuphala varna. Bhasma* was 95% *rekhapurna* and 75% found positive in *varitara* test.

Batch "C" was also prepared same as batch "A". After 1st *puta* colour of pellets was black. Surface of pellets was rough and cracked. After triturating again powder was converted into black colour. After 2nd *puta* some particles of *Kanta lauha* was remain as such which are not converted into powder form. Surface of pellets were rough. After *puta* colour of pellets was bluish black but surface of some pellets was brown in colour. After 5th *puta* colour of pellets was reddish brown and surface of pellets was rough. 20% *bhasma* was *rekhapurna*. After 10th *puta* Pellets were little hard then previous. Colour of pellets was turned to reddish black. Surface of pellets was smooth and 45% *bhasma* was *rekhapurna*. After 15th *puta* Pellets were little hard. Colour of pellets was slightly blackish red. *Bhasma* was became 60% *rekhapurna* and 50% *bhasma* was found positive in *varitara* test. After 18th *puta* Pellets were now soft and easily breakable by hand. Colour of *bhasma* was blackish red i.e. *pakvajambuphala varna. Bhasma* was 95% *rekhapurna* as well as 75% *bhasma* was found positive in *varitara* test.

Characterization

The drug obtained in the three batches was characterized according to the ancient parameters as well as modern methods. The ancient parameters are *Varna, Rasa, Sparsha, Rekhapurnatvam, Varitaratvam, Nirdhumatvam, Nischandratvam*. A portion of powders obtained in each batch was also observed in Field Emission Scanning Electron Microscopy (FESEM) (Quanta 200 FEG, FEI) in Environmental mode for understanding the microstructure. Energy dispersive X-ray analysis was also carried out in-situ to know the chemical constitution. Chemical constitutions of the powders were also determined independently by X-ray Fluorescence

Spectroscopy. Different Phases and their crystallographic forms present in the *bhasma* were identified by Fourier Transformed Infrared Spectroscopy (FTIR).

Ancient parameters

Modern parameters

This analysis includes

a) Loss on drying.
b) Determination of Ash value.
c) Determination of Acid insoluble
d) SEM (Scanning Electron Microscopy) and EDAX (Energy dispersive X ray analysis) Analysis
e) FTIR Analysis

Loss on drying

This test was performed to find out the moisture content in the sample. One gram of exactly weighed sample was taken in a previously weighed petri dish from each batch and dried in an oven at 110°C till the same acquired constant weight. Then the petridish was taken out, weighed after self cooling and from the weight loss the percentage of loss on drying was calculated and expressed as %w/w9.

Property	Batch A	Batch B	Batch C
Loss on drying (% w/w)	0.30	0.35	0.35

Ash value

This test was conducted to assess the total ash content of the sample. It was determined by incinerating about 2 g of accurately weighed sample in tarred silica crucible in an electric muffle furnace at 700°C, then allowed for self cooling and weighed. The percentage of ash was calculated and expressed as %w/w

Property	Batch A	Batch B	Batch C
Ash value (% w/w)	99.10	98.50	98.50

Acid insoluble ash

This test was carried out to assess the percentage of acid insoluble inorganic content of the sample. The ash obtained in process of determination of ash value was boiled for 5 minutes with 50 ml of dilute hydrochloric acid, the insoluble matter was collected on ash less filter paper, washed with hot water and ignited to get constant weight. The percentage of acid insoluble ash was calculated and expressed as %w/w.

Property	Batch A	Batch B	Batch C
Acid insoluble ash (% w/w)	25.50	15.20	15.10

FESEM

The samples in the present study being mixtures of oxides normally require conductive coating. With availability of environmental mode in the FESEM used for the study the *bhasma* powders were sprinkled on an adhesive carbon tape and observed directly or were compacted under light pressure if required and observed.

Teekshna lauha

Fig.1(a) shows a microstructure of iron turnings, the raw material, with typical metallic appearance. EDAX analysis shows high iron percentage with small amounts of Al and Mn typically present in the raw material(Fig. 1(b)).

Scanning electron microscopy of Sthalipaka samples showed clear grains of the iron. It could be inferred from the dark contrast that most of the surface is free from oxide; except a small volume fraction of oxide in the form of white particles. EDAX analysis revealed presence of a high percentage of carbon, which could be due to the organic material coming from the addition of *Triphala kwatha* in *Bhanupaka* and *Sthalipaka* steps.

The first *puta* samples exhibited complete coating of oxide particle (inferred from the bright contrast of the

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particles due to charge accumulation under the electron beam). By tenth *puta* such surface coating was more abundant. Two types of particles were noticed at this stage. The larger particles of dimension 10 to100µm range had higher percentage of Fe (58.72wt.%), O (19.76wt.%), K (6.14wt.%) as revealed by EDAX in Fig.1(c). This lump may be agglomerated iron oxide and potassium salt(coming from trituration with *kwatha*.) Fig 1(d) shows lumps of medium size as well as fine individual particles after 20th *puta.* Lumps are in size range 2-4 µm and individual particles in the range of 100-500nm.

Kanta lauha

Fig.2(a,b) show the micrographs of crushed powder of *Kanta lauha* of the two batches (B1 & C1). The angular particles of the mineral can be viewed clearly in these micrographs. Contrast differences between different sets of particles could be due to the difference in chemical constitution. Those having very bright appearance(a) are less conducting and are thus showing charge build – up while those with gray contrast(b) are more conducting and could be pure magnetite particles (*Kanta lauha*). The brighter particles could be other oxides which constitute the gangue material. EDX analysis of the particles shown in Fig.2(c,d) confirms the chemical constitution of the raw material.

When subjected to *puta* process the *Kanta lauha* is undergoing a purification process and the gangue material of various other oxides present along with iron oxide are getting separated and are deposited on the surface as shown in Fig.2(e,f) by fifth puta, such surface coating is more evident as in the bright contrast present.

Fig. 3(a,b) shows the SEM micrographs of the powder obtained after 18th *puta* corresponding to both batches of *Kanta lauha.* Here the particles can be noticed to have a coating of the oxides which renders them a brighter appearance due to charge build up. Particle size in case of both samples of *Kanta lauha bhasma* is 100-300nm in range.

FTIR

It becomes essential to know the number and nature of oxides that are forming during the *puta* process, more so in the case of *Kanta lauha* samples, where Fe is present in oxidized state to begin with. Therefore, FTIR spectroscopy was carried out for all samples. Individual spectra are obtained at each stage of preparation. Table 4 shows characteristic absorption peaks corresponding to Magnetite, Hematite and Meghamite. On the basis of this data the spectra obtained at various stages of processing could be interpreted to consist of peaks corresponding to Magnetite, Hematite and Meghamite at *sthalipaka* stage and only Meghamite and Hematite in sample subjected to increasing number of *putas*10. Only three of these spectra are shown in Fig.4. In (a) a typical spectrum from Meghamite is depicted. FTIR spectrum of the raw material *Kanta Lauha* is depicted in (b) where a prominent peak due to magnetite can be noted. Additional peaks in this plot are due to the impurities in ore samples(known as gangue material). Fig. 4(c) shows the spectrum of the *bhasma* after 18 *Puta* (Batch-C) with a prominent peak corresponding to Fe2O3 at around 530 cm-1. Data corresponding to important forms of iron oxide are presented in the Table mentioned below..

Table 4: Peak positions in FTIR corresponding to various forms of iron oxide.

S. No.	Elements	Corresponding Peaks
1.	Hematite	620, 540, 460
2.	Magnetite	580, 400
3.	Meghamite	690, 635, 560, 440, 420

EDAX Analysis

The EDAX analysis of Batch A (iron turnings) showed 91.26% of iron and small amount of other materials like Mn, Al etc. in raw material, later on with increasing number of *putas*, the iron percentage was found to decrease. The EDAX analysis of Batch B (magnetite) shows a higher percentage of iron i.e. 94.49% and oxygen is 5.51%. After *puta* process, various elements which were not present in the initial raw material showed up in the analysis. These may be coming from *triphala kwatha* used during trituration after each *puta*. Analysis of lauha bhasma after final *puta* (see Fig.1(c)) showed the iron content to be 92.45% which is only marginally less than the initial value. The EDAX analysis of Batch C (magnetite) shows,Fig. 5, higher percentage of iron i.e. 94.66%, which decreased during the process of *bhasmikarana* and reached to 89.84% after final *puta.* Other elements increased in their percentage as earlier in the case of Batch A and B. In all the above analyses iron is present in the combined form of oxides except in the case of raw material of Batch A.

Discussion

Analytical study was carried out with an intention of understanding the effect of process parameters on the method of drug manufacturing and the difference in the final product, if any, between different starting materials chosen.

Loss on drying of lauha bhasma was 0.30, 0.35, 0.35 for batch A, B, C respectively. The ash value of lauha bhasma was 99.10, 98.50, 98.50 for batch A, B, C respectively. This ash value of lauha bhasma shows that there is very little evaporative substance. Acid insoluble ash of lauha bhasma was 25.50, 15.20, 15.10 for batch A, B, C respectively.

From the analytical study it is clear that Fe present in the elemental form along with its impurities in the turnings is converted to two different forms of iron oxide viz., Meghamite and Hematite6. Other impurities such as Si, Al etc are also oxidized. In the case of *Kanta lauha* (Magnetite), where Fe is present in oxide form Fe_3O_4, it is transformed to higher oxide forms. Such a reaction is possible7 during the *puta* process at as low a temperature as 3750C according to this reaction

$2Fe3O4 + 1/2\ O2 \longrightarrow 3\ \alpha\text{-}Fe2O3$ (431 Jg-1)

The minor elements present either as impurities in the metal (Batch-A) or as impurities in the mineral magnetite (*Kanta lauha*) also get oxidized (Batch-B) and remain with the *Bhasma.* Thus these are also detected in the chemical analysis. Further, the two crystallographic forms in which the Fe2O3 iron oxide can be present are γ–Fe2O3 (Meghamite) and α–Fe2O3(Hematite). Both crystallographic forms were noticed (see Fig. 4) in the samples after completion of *Putas.* Thus one observes that the *puta* is a process of oxidation of the metal(Batch-A) or the lower oxide of Fe in *Kanta Lauha* to higher oxides. Apart from these the chemical constituents of *Triphala,* which is an essential ingredient of the mixture for *puta* and also for trituration between *putas,* consists of a host of minor elements such as Na, K, Mg, Ca, Cl and P and in trace quantities twenty three other elements such as AI, Ba, Br, Cd, Co, Cr, Cs, Cu, Fe, Eu, Hf, Hg, La, Mn, Ni, P, Pb, Rb, Sb, Se, Th, V and Zn elements8. These get oxidized during *puta* and remain as integral part of the final *Bhasma.* These may be removed, or in other words iron oxide might get selectively picked up, during preparation of a "*Lohasava*"2. The Ayurvedic method of preparation of *Bhasma* is a combination of wet-chemical and pyrometallurgical method of preparation. Thus, the impurities will remain with the prepared material unless otherwise, the oxides so formed during *puta* are volatile (e.g. Silver oxide in sample C) and are removed

during *puta.* In the conventional method of metallurgical extraction of iron from its ores, the minor impurity elements such as Si, Al, Ca, Mn from the mineral are removed in the form of slag during the pyrometallugical method (8).

Conclusions

Loss on drying, ash value and acid insoluble ash results shows that the little evaporate substance only present in lauh bhasma indicating its best quality.

In the case of tikshna lauha the iron is converted to two different forms of iron oxide viz., Meghamite and Hematite. In the case of *Kanta lauha* (Magnetite), where Fe is present in oxide form Fe3O4 , it is transformed to higher oxide forms. It is also observed that the chemical constituent of triphala consists of a host of minor elements and remain as integral part of the final bhasma.

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Table 1 Observations of *Samanya Sodhana*

S. No.	Batch	Colour		Consistency		Averg. Time taken for maximum heating (min)	Averg. Temp. of material (0C)	Wt. before *Sodhana* (g)	Wt. after *Sodhana* (g)
		B. S.	A. S.	B. S.	A. S.				
1.	A	Blackish white	Black	Hard	Brittle	20-25	900	500	540
2.	B	Grayish black	Black	Hard	Brittle	15-25	900	500	520
3.	C	Grayish black	Black	Hard	Brittle	15-25	900	500	510

Table 2 Observations of *Visesha Sodhana*

S. No.	Batch	Colour		Consistency		Averg. Time taken for maximum heating (min)	Averg. Temp. of material (0C)	Wt. before *Sodhana* (g)	Wt. after *Sodhana* (g)
		B. S.	A. S.	B. S.	A. S.				
1.	A	Black	Dark black	Brittle	More brittle	15-20	900-950	520	610
2.	B	Black	Dark black	Brittle	More brittle	15-20	900-950	500	530
3.	C	Black	Dark black	Brittle	More brittle	15-20	900-950	480	510

B. S.- Before *sodhana,* **A. S**.- After *sodhana*

Table 3. Analysis of *Lauha bhasma* by ancient method

S. No.	Test	Sample A	Sample B	Sample C
1.	*Varna*	*Pakvajambuphala varna*	*Pakvajambuphala varna*	*Pakvajambuphala varna*
2.	*Rasa*	Tasteless	Tasteless	Tasteless
3.	*Sparsha*	*Mrudutva* and *shlakshnatva* was felt by simple touch with finger tips	*Mrudutva* and *shlakshnatva* was felt by simple touch with finger tips	*Mrudutva* and *shlakshnatva* was felt by simple touch with finger tips
4.	*Rekhapurnatvam*	The *Lauha bhasma* was rubbed in between index finger and thumb. It enters into the furrows of the finger- Positive	The *Lauha bhasma* was rubbed in between index finger and thumb. It enters into the furrows of the finger- Positive	The *Lauha bhasma* was rubbed in between index finger and thumb. It enters into the furrows of the finger- Positive
5.	*Varitaratvam*	A small amount of *Lauha bhasma* was	A small amount of *Lauha bhasma* was	A small amount of *Lauha bhasma* was

		carefully sprinkled on the water. It was found that 75% *bhasma* was floating on the water surface- Positive	carefully sprinkled on the water. It was found that 75% *bhasma* was floating on the water surface- Positive	carefully sprinkled on the water. It was found that 75% *bhasma* was floating on the water surface- Positive
6.	*Nirdhumatvam*	The *Lauha bhasma* was sprinkled on red hot coal. It did not emit smoke- Positive	The *Lauha bhasma* was sprinkled on red hot coal. It did not emit smoke- Positive	The *Lauha bhasma* was sprinkled on red hot coal. It did not emit smoke- Positive
7.	*Nischandratvam*	It was not having any luster positive	It was not having any luster positive	It was not having any luster positive
8.	*Apunarbhava*	The *Lauha bhasma* was triturated with *Gunja, Ghrita, Madhu, Tankana* and *Guggulu* and made into pellets. Then it was subjected to puta on 6000C in EMF maintained for one hour. Next day after *swangshitala* pellets were triturated and no any agglomeration was found.	The *Lauha bhasma* was triturated with *Gunja, Ghrita, Madhu, Tankana* and *Guggulu* and made into pellets then it was subjected to puta on 6000C in EMF maintained for one hour. Next day after *swangshitala* pellets were triturated and no any agglomeration was found.	The *Lauha bhasma* was triturated with *Gunja, Ghrita, Madhu, Tankana* and *Guggulu* and made into pellets then it was subjected to puta on 6000C in EMF maintained for one hour. Next day after *swangshitala* pellets were triturated and no any agglomeration was found.
9.	Physical test specific to *Kanta Lauha*	-	Nimba patra kalka was put over the kanta lauha (Magnetite iron ore) pieces and left for 24 hours, next day it was observed in which bitterness of nimba patra kalka decreased, when hingu was placed over Kanta lauha, its odour also decreased.	Nimba patra kalka was put over the kanta lauha (Magnetite iron ore) pieces and left for 24 hours, next day it was observed in which bitterness of nimba patra kalka decreased, when hingu was placed over Kanta lauha, its odour also decreased.

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