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BIOPROCESS VARIABLES OF MAGNETITE NANOPARTICLES USING MODIFIED MODERN BHASMIKARAN METHOD

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ABSTRACT

Lauha bhasma or iron oxide is a well known ancient form of Ayurvedic medicine for treating diseases such as anemia and jaundice since seventh century BC. The preparation generally involves a tedious long process of conversion of raw materials into ash or bhasma for this purpose. In this paper, a novel and modern form of Ayurvedic method has been used to prepare iron oxide nanoparticles which is both cost effective and eco-friendly. The particles were synthesized through multiple filtration and calcination steps resulting in fine powders which were then analyzed through XRD, TEM and VSM for their structural, morphological and magnetic properties which showed a small transition from hematite phase to magnetic magnetite phase post to the final filtration technique.

Keyword: Ayurveda, Lauha bhasma, Iron oxide nano particles.

INTRODUCTION

Bhasma or ash is an integral part of Ayurveda, a science that describes about usage of metals and minerals at various purities and concentrations for the treatment of chronic disorders. Bhasmas have recently acclaimed to be metallic nanoparticles prepared by green Ayurvedic preparations by treating raw materials with herbal juices or decoctions. Lauha bhasma or iron ash is one such Ayurvedic preparation that has shown effective cure towards diseases such as hyperlipidemia, tuberculosis, urinary tract infections, obesity etc, among Indians[1]. Lauha bhasma is also known to have shown useful application in various fields such as magnetic resonance imaging, drug delivery and hemoglobin regulation[2]. The Preparation of Lauha bhasma is a tedious and time consuming method which involves five major steps namely, samanya sodhana (general purification step), visesha sodhana (special purification step), Bhanupaka (reaction under sunlight), sthalipaka (roasting of contents in iron vessel), and puta (calcination) when it comes to the ancient method[3]. These ancient methods though effective, are scientifically vulnerable without proper understanding of the process and quantitative and qualitative reports at each stage. Although a part of this work has been discussed in our earlier paper[4], here we discuss in detail with respect to the change in structural and magnetic properties of the prepared Lauha bhasma particles. In this paper, a more scientific and modern approach towards the formation of Lauha bhasma has been discussed where all the samples have been synthesized under standard laboratory conditions and were analyzed using known analytical techniques such as X-ray diffraction (XRD) system, transmission electron microscope (TEM), and vibrating sample magnetometer (VSM). The formation of multiple phases during this process was observed and reported in this paper.

MATERIALS AND METHODS

The preparation of the particles is discussed elsewhere[4] in our previous publication in detail. The preparation of the particles mainly involves the usage of an iron precursor (iron filings also known as Lauha Chuma) and a set of treatment liquids such as sesame oil (used for removing grease and rust from lauha chuma), butter milk (used for removing oil), rice gruel (contain phytic acid to remove Fe^{3+} ions)[5], Panchakavya (antibacterial agent)[6], horse gram powder (contain gallic acid, remove Fe^{3+} ions), triphala (contain equal combination of *Phyllanthus emblica, Terminalia chebula, Terminalia bellerica*). Each of these carry a specific type of property as mentioned above with a special property of reducing Fe^{3+} ions to Fe^{2+} ions[7]. Iron in the divalent state can enter mucosal cells easily. These liquids treatment were used in the process of preparation with specific steps involving cleaning and filtration followed by drying or calcination.

Preparation Steps

Step 1: Normal Purification

The normal purification step was done by quenching iron filings (40 g) in sesame oil (20 ml) butter milk (20 ml) and rice gruel (20 ml) each for 30 minutes. This process was repeated 7 times before the iron filings were fired at 560°C for 30 minutes. This process makes sure that none of the grease or other contaminants attached to the iron ore are present during further processing.

Step 2: Special Purification

The product of the normal purification step was then left for immersion in panchakavya (20 ml) overnight (24 hours) before it was cleaned with the same treatment liquid 6 times. Cleaned product was mixed with 2 g tripahal decoction and re-immersed in panchakavya for 2 hours. Post immersion, panchakavya was decanted from the iron ore and the iron ore was exposed to ultraviolet radiation for a period of 24 hours.

Step 3: Heat Treatment

Post ultraviolet radiation exposure, tripahal decoction (3 g) was added to the above product and heated at a temperature of 100°C. Another 0.5 g of tripahal decoction was added to the product before it was fired at a temperature of 900°C for 2 hours. The treated particles were then filtered using a 100 nm pore size filter in order to obtain nanoparticles of the obtained product.

Characterization techniques

The obtained product was studied for its structural properties by D8 Advance Bruker XRD with CuKα source to between 2θ values 20° and 80°. TEM was done for the samples using a FEI company TECNAI model.

RESULTS AND DISCUSSION

XRD Analysis

XRD patterns were taken for samples at every stage of purification and sintering. This was done in order to keep a track of the change in crystal structure with each proceeding step. All the XRD patterns can be seen in the Figure 1 and 2 with subdivisions a to f (Figure) describing each of the purification and sintering steps. The initial iron filings that are used are usually a mix of various iron compounds and grease. After the first purification step with sesame oil, the XRD pattern of the iron filings shows a clear hematite phase formation. This is due to the intrinsic nature of iron to stably form a rhombohedral trivalent ionic state in its natural state. No change in the XRD patterns of samples taken after purification with butter milk (XRD patterns seen in subdivisions b). This can be attributed to the fact that the function of this treatment liquid was to cleanse the iron filings off dirt and grease rather than change any of its intrinsic structural properties.

The XRD patterns c, d and e were taken after the samples were treated using rice gruel, panchakavya and tripahal decoction respectively. Here a change in the XRD pattern can be seen wherein the relative intensity of the peak at 2θ values 33.4 starts to decrease coupled simultaneously by increase in the peak intensity at 2θ value 35.5. These patterns when matched with JCPDS data gave higher intensity match towards presence of magnetite phase. This can be attributed to the fact that iron chelates such as phytic acid (rice gruel) and gallic acid (tripahal decoction) which not only capture ferric ions but also enhance the formation of ferrous ions in any material. This particular property of the treatment liquids could have induced the formation of higher amounts of ferrous ions in the iron filings thereby increasing the formation of magnetite.

Further heating at 900°C (XRD pattern in Figure 1f) in the presence of tripahal decoction makes sure that the formation of magnetite is in higher progress rather than hematite. Magnetite structure involves the formation of a complex inverse spinel structure rather than rhombohedral structure as seen in the case of hematite. Magnetite contains two ionic states of iron with ferrous ion occupying one of the two rhombohedral sites while each of the ferric ions occupy a tetrahedral and an octahedral site inside the inverse spinel lattice.

From the XRD data, the crystallite size was calculated to be 30 nm using the Scherer's formula.

TEM Analysis

The TEM images were taken from the samples that were filtered through the 100 nm filter paper after the final sintering was done. From the TEM images in Figure 2, the particle size of the powders that were obtained can be confirmed as approximately 20 nm in size. The particle size distribution is almost equal with most of the particles in spherical shape. The crystallite size analysis from the XRD results stand strong with the particle sizes measured using the TEM showing that the each of the synthesized particles are single crystal in nature.

VSM Measurement

The VSM measurements were done on 2 sets of samples. The sample B was the iron filings that were purified using sesame oil and butter milk, while the samples F was the one which was filtered after the final sintering process. In the first sample, the formation of a small hysteresis loop with magnetization value of 3.4 emu/g was seen. This low magnetization and presence of tripahal value is due to the crystal structure and particle size of the material being tested. In the first sample, from the XRD results, it is evident that only hematite phase is present. Hematite is known to be a weak ferromagnetic material with its ferromagnetic property arising from the canted antiferrromagnetic arrangements inside its structure which is shown in Figure 4. The particle sizes in the case of the first sample are very large as they were neither filtered nor powdered in any sense resulting in particles with diameters larger than the critical super-paramagnetic size.

In the second sample, the VSM graph clearly shows the formation of superparamagnetic particles which have high susceptibility values and no remnant magnetization corresponding to them. This can be attributed again to the crystal structure and the particle sizes of the samples. The presence of magnetite phase in the sample gives a high magnetization value of 15.18 emu/g while the presence of particles smaller than 20 nm in size yields a superparamagnetic behavior in the sample.

CONCLUSION

A novel process for the preparation of ferric ion quenched magnetite has been shown in this paper with only iron filings as precursors. The ancient bhasmikaran process for the formation of iron ash in the form of hematite was modified in this case to form a completely different magnetic phase (magnetite). The process was analytically monitored throughout the preparation steps using XRD which yielded an important finding that $Fe3+$ quenchers such as rice gruel and triphala play a major role in changing the phase of hematite to magnetite even at high temperatures. The TEM and VSM results taken for the final sample showed a homogeneous particle size and superparamagnetic nature giving a hint towards a simple and green synthesis process for the preparation of magnetite for biomedical applications.

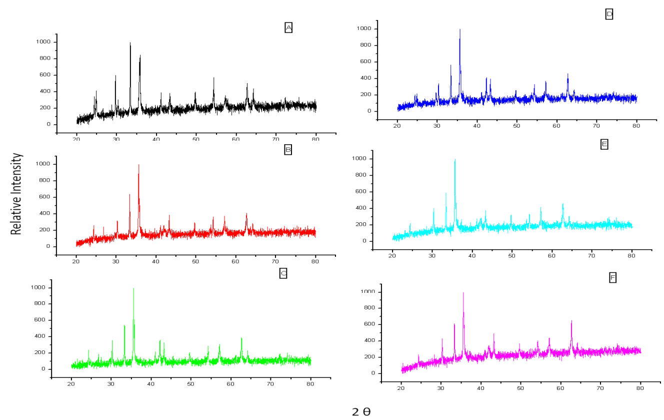

Figure 1: XRD spectrum of iron oxide nanoparticles

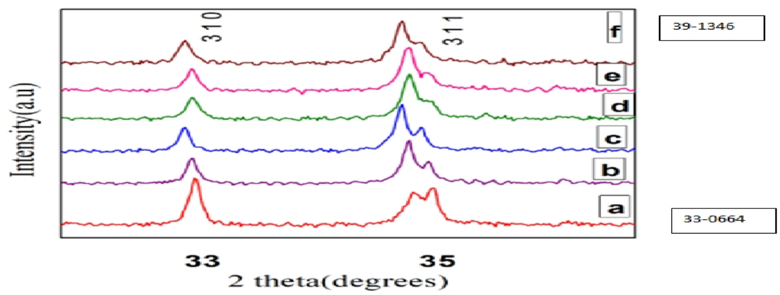

Figure 2: Peak shifting from α to γ Fe_2O_3

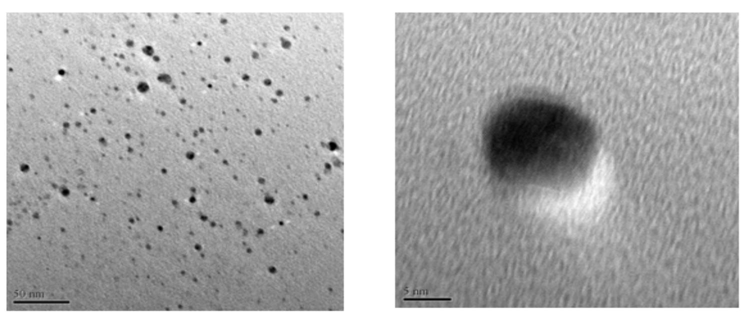

Figure 3: TEM images of Lauha bhasma sample

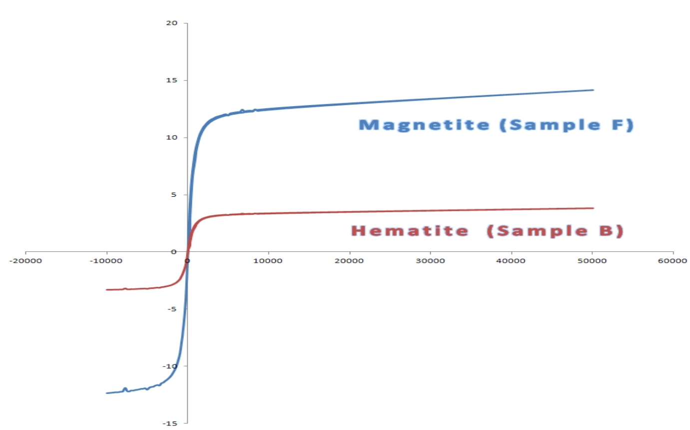

Figure 4: VSM of samples taken after normal purification and final calcinations process

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