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Research article

A Green approach for the synthesis of nano-sized iron oxide, by Indian ayurvedic modified bhasmikaran method

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Abstract

Lauha bhasma (iron oxide) is used extensively in Ayurvedic literature for management of various diseases like anemia, jaundice etc., as iron is very hard metal, it has been converted into bhasma (herbo metallic preparation) for medicinal use. Since seventh century BC twenty bhasmas were analyzed and widely recommended and used for curing various chronic ailments. In this paper, a novel modern ayurvedic method for synthesizing ayurvedic medicine iron oxide nano particles which is cost effective and ecofriendly technique was reported. The synthesis of iron oxide nano particles have been done using iron filings as a precursor material, subjected to normal and special purification to obtain the final product as Lauha bhasma. X-ray diffraction was used to monitor phase transformation from α - to γ- phase and characterizations were done using Thermo gravimetric analysis, Transmission electron microscope, Fourier Transform Infrared Spectroscopy.

Keywords: Ayurveda, Lauha bhasma, herbo metallic preparation, Iron oxide nano particles.

1. Introduction

Bhasma is an integral part of Ayurveda describes about using metals & minerals for chronic disorders in various combinations, dosages at various levels of purities. Bhasmas are unique Ayurvedic metallic preparations treated with herbal juices or decoction and these are claimed to be biologically produced nanoparticles. In most of the developing countries like India, anemia is considered to be most common disease. Due to tremendous properties Lauha bhasma is applied in various fields such as MRI, drug delivery, curing anemia, hemoglobin regulation [1]. Size-induced structural phase transformation of α to γ Fe_2O_3 has been reported and the observations on samples with narrow size distributions and different mean sizes of iron oxide showed that α to γ Fe_2O_3 phase transformation occurred these transformations are mainly due to diffusion, alternation of phase composition, production of metastable phases by consecutive purification steps. Synthesis of iron oxide nano particles by ancient method is tedious, but by using modern laboratory techniques has become successful.

2. Materials and Method

The raw material used for the synthesis of Lauha bhasma is Iron filings; treating liquids for purification are sesame oil (used to remove greasy material, rust from the material), butter milk (removes oily matter and impurities), rice gruel (contain phytic acid and remove Fe3+ ions , panchgavya (antibacterial agent), horse gram powder (contain gallic acid, remove Fe3+ ions), triphala (contain equal combination of Phyllanthus emblica, Terminalia chebula, Terminalia bellerica). By all the purification steps Fe^{3+} ions get reduced to Fe^{2+}, iron in the divalent state enter mucosal cells easily into the body.

2.1 Preparation of Lauha bhasma (iron ash)

The raw material Lauha churna (iron filings) was taken as raw material for the synthesis process. The preparation involves mainly two steps- normal purification, special purification, in which modern synthesis technique is used to produce final product, Lauha bhasma [2-3].

2.2 Normal Purification

Normal Purification process involves three sub steps: In the first step of purification process, 40 grams of iron filings were taken in a beaker, using 20 ml of sesame oil as quenching medium, immerse for 30 minutes and filter by filteration setup. Then so obtained filtrate is heat treated at 530-560°C for 30 minutes by which the filtrate is completely dried. Each of the quenching process was repeated for seven times with each treating liquid, by using fresh medium every time.

2.3 Special Purification

The product obtained from normal purification is taken in a beaker and immersed in 20 ml of panchgavya for 24 hours. Then wash with panchgavya for 6 times by filteration. Add 2 grams of triphala decoction and again immerse in panchgavya for 2 hrs. The product obtained from special purification is exposed to sunlight for 24 hours.

2.4 Heat treatment

Finally the product is heat treated in iron pan. Add 3 grams of triphala, at 95-100°C to dry for 1hour. Again add 0.5 grams of triphala, at 900°C to dry for 2 hours then the final product obtained is Lauha bhasma (iron ash).

3. Characterization Techniques

X-ray diffractometer (D8 Advance Bruker, Germany) with Cu Kα was used to study the crystallographic phases. Thermal analyses of the samples were done with TG-DTA (EXSTAR TG/DTA 6000 series). Fourier Transform Infrared Spectroscopy (FTIR) was performed to identify types of chemical bonds, i.e. functional groups in a molecule (Model no: Perkin Elmer precisely FT-IR spectrometer) over the wave number range of 4000-500 cm^{-1}.

4. Results and Discussion

4.1 X-Ray diffraction analysis:

Iron oxide nanoparticles obtained from modern synthesis through ayurvedic procedure was examined by XRD. By using raw data file we can find average crystallite size of the sample. The crystallite size of the powders was calculated from an X-ray diffraction peak broadening using Scherrer formula. The crystallite domain size was calculated from the width of the XRD peaks, assuming that they are free from non-uniform strains, using the Scherrer formula [4].

$$t=0.9\lambda /\beta \cos \theta \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where, β is the full width at half maximum (FWHM) of the XRD all peaks, t is the crystallite size, λ is the wave length of the X-ray, θ is Bragg's angle.

In the figure 1 XRD graphs represent the sequential purification steps in which graph (a) indicates treatment with sesame oil, graph (b) indicates treatment with starch solution, graph (c) indicates treatment with butter milk, panchgavya, horse gram decoction, starch solution, graph (d) indicates treatment with triphala, graph (e) indicates evaporation under sunlight, graph (f) indicates final product Lauha bhasma (iron ash).

In this figure 2, phase transformation is clearly seen i.e., the first purification step-(a) highest peak is at 33° indicating α-phase of Fe_2O_3compared with the standard JCPDS data (card no. 33-0664) contain hkl planes at (2 2 0), (1 0 4), (1 1 0), (2 0 2), (0 2 4), (1 1 6), (0 1 6), (2 1 4). But in (b) the highest peak is shifted towards 35° indicating

γ-phase of Fe2O3 compared with the standard JCPDS data (card no.39-1346) [5] contain hkl planes at (2 2 0),(3 1 0),(3 1 1),(4 0 0),(4 2 1),(4 3 0),(5 1 1),(4 4 0). In the further graphs γ-phase remains constant and no other phase transformation is observed. As the purification steps are increasing the peaks appears to be sharper and narrower, phase transformation is clearly observed.

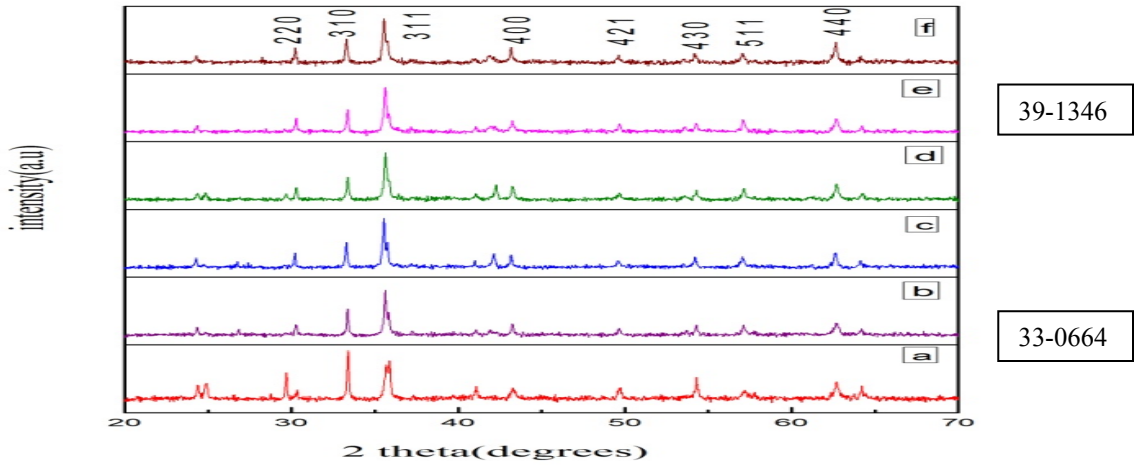


Figure 1: XRD spectrum of iron oxide nanoparticles

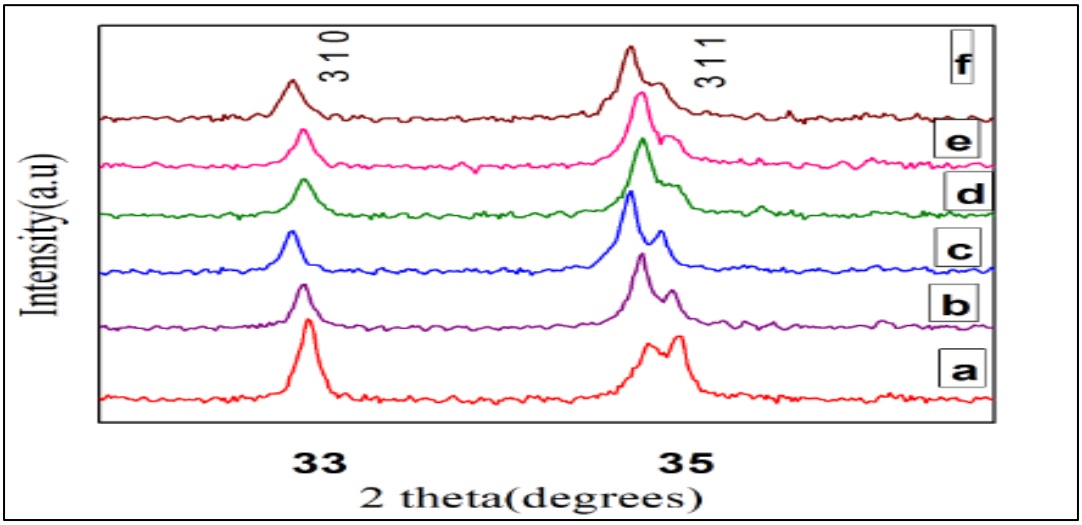


Figure 2: Peak shifting from α to γ Fe_2O_3

4.2 Fourier Transform Infrared Spectroscopy (FTIR):

FTIR spectra with wavenumber ranger from 450-4000 is shown in figure 3 for the final Lauha bhasma sample indicates three vital peaks, the spectra displays broad absorption around 3733.83 cm^{-1} was assigned as OH stretching, H-O-H bonding at 1632.56 cm^{-1} and the main Fe-O stretching was observed at 577.94 cm^{-1} indicating the presence of iron oxide nanoparticles [5].

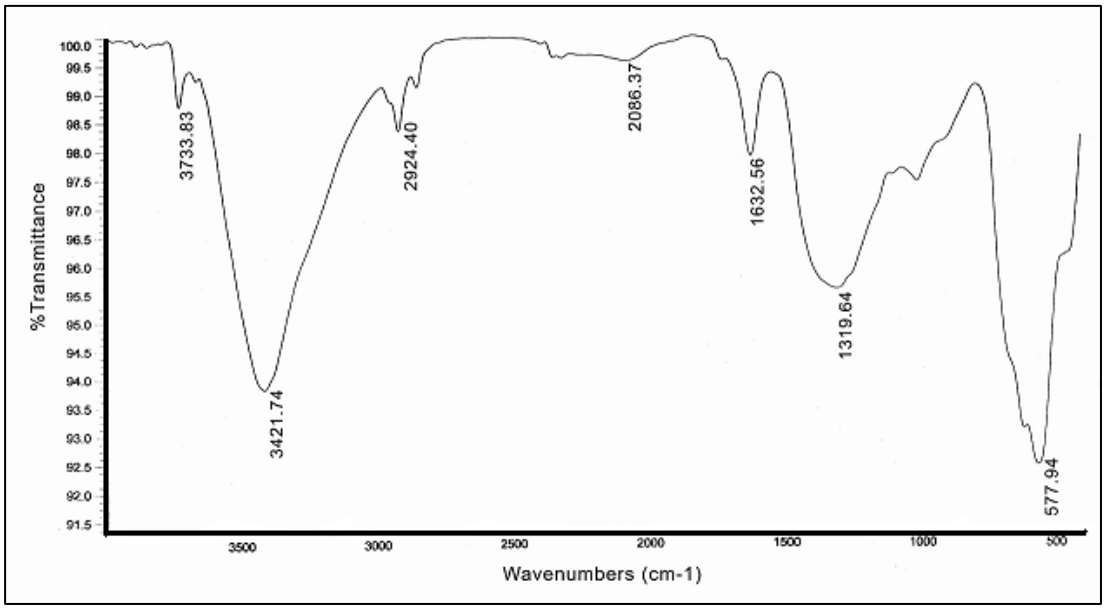


Figure 3: FTIR spectra of Lauha bhasma sample

4.3 Thermo Gravimetric and Differential Thermal Analysis:

TG & DTA curves of Fe_2O_3 nanoparticles are as shown in Figure 4. The temperature range is 30°C to 800°C. In TG analysis, the total weight loss percentage of the sample is 1.3674%.The initial weight loss observed below 300°C, it corresponds to the liberation of adsorbed moisture on the surface of the sample. The weight loss is 0.7126%.At transition range of temperature 300-500°C; the weight loss is 0.3939%. It corresponds to the elimination of carbon group compounds. In the final stage, temperature range from 500-800°C; the weight loss is about 0.2603%. This corresponds to phase transformation from Fe to Fe_2O_3.

From DTA, 3 exothermic and one endothermic peak is observed. At 600°C to 700°C both the endothermic and exothermic peaks are observed due to phase transformation. It corresponds to the complete liberation of carbonaceous and other inorganic materials in the sample and it infer that the sample has very extreme purity and very small weight loss.

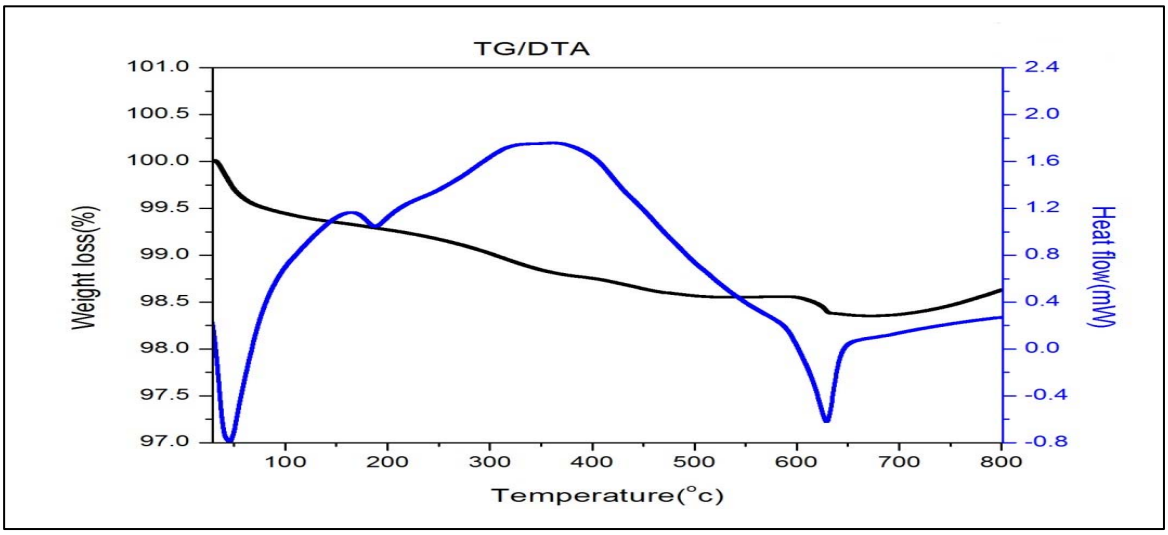
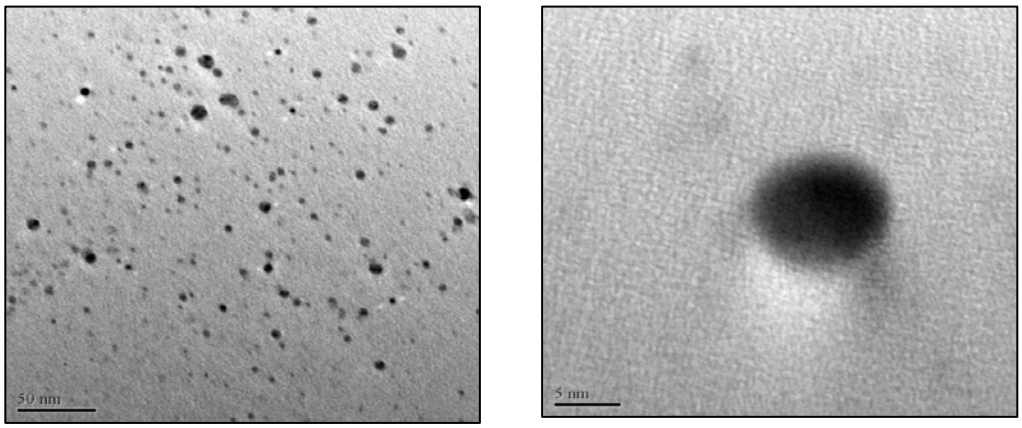


Figure 4: TG/DTA graph of Fe_2O_3 sample

4.4 Transmission Electron Microscopy:

From figure 5a, the micro graph shows the dispersion of nanoparticles. By calculating crystal structure at 50 nm scale, we obtained around 20 nm nanoparticles are indicated. From figure 5b, the pattern of TEM analysis indicates the presence of spherical structure of iron oxide nanoparticles which is in good agreement with that estimated by Scherrer formula based on XRD pattern [6].



a b

Figure 5: TEM images of Lauha bhasma sample

Conclusion

The modern method of synthesizing Lauha bhasma (iron oxide nano particles) was successful, which was observed from XRD. By undergoing normal and special purifications the sample was tuned into fine nano crystallite. We have investigated structural, thermal properties. X-ray diffraction was used to monitor phase transformation from α - to γ-phase that indicates the face-centered cubic structure for iron oxide nano particles, which is good agreement with TEM and FTIR functional spectrum. TG/DTA represents very low percentage of weight loss indicates high purity in the sample. TEM represents well dispersion and perfect cubic structure of the sample.

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Reference

[1] Pandit S, Biswas TK, Debnath PK, Saha AV, Chowdhury U, Shaw BP, et al.: Chemical and pharmacological evaluation of different ayurvedic preparations of iron. J Ethnopharmacol 1999, 65:149-56.

[2] Balaji Krishnamachary et al, Rajan K.Sekar: Scientific validation of the different purification steps involved in the preparation of an Indian Ayurvedic medicine, Lauha bhasma. Journal of Ethanopharmacology 2012, 142(1): 98-104.

[3] Singh Neetu, Reddy K R : Particle size estimation and elemental analysis of Lauha Bhasma, IJRAP,2011 2(1): 30-35.

[4] B.D Cullity, Elements of X-Ray Diffraction, Addison-Wesley Publishing Company, Inc., Philippines (1978)

[5] Ruipeng Fu, Wei Wang, Rongjiang Han, Kezheng Chen: Preparation and characterization of γ–Fe_2O_3/ZnO ccomposite particles. Materials Letters 62 (2008) 4066-4068

[6] Mohammad Kooti, Layla Matturi: Microwave-Assisted fabrication of γ-Fe_2O_3 Nanoparticles from Tris (acetylacetonato) Iron (III) Int .NanoLett., Vol.1,No.1,January 2011:pp.38-42