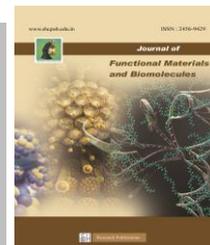




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## Investigating the Effect of Calcination on the properties of Iron Oxide Nanoparticles

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### Abstract

Over the past decades, nanomaterials have been largely focused as they shows distinctive size dependent characteristics. Sonicator has been exploited to synthesize iron oxide nanoparticles. The as prepared and sample calcinated at 400°C were characterized using (SEM) reveals the morphology of the prepared sample. The XRD studies reveals the formation of iron oxide nanoparticles with rhombohedral structure. The FTIR and UV-Visible studies are used to identify the vibrational groups and absorption range of the prepared samples. Calcinating the sample found to increase the crystallinity of the sample.

**Keywords:** Nanomaterials, Iron oxide, SEM and XRD.

### 1 Introduction

The magnetic nanoparticles exist many important magnetic properties such as high coercivity, high magnetic susceptibility, low Curie temperature and super paramagnetic etc [1]. Magnetic nanoparticles possess a wide interest among the researchers due to its broad range of applications in data storage, catalysis, bio-applications and magnetic fluids.

Iron oxide, a noble magnetic oxide occur in many forms with maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ , magnetite ( $\text{Fe}_3\text{O}_4$ ) [2], and hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) being probably the most common. They have possessed a different behavior in accordance with the size of the magnetic material [3]. When the size of the material is sufficiently small it behaves as paramagnetic and whereas, the grain size is in micrometer range the material shows ferromagnetic properties [4]. The varying size of the material also possesses to change the application of the encapsulated particles [5]. When the size is reduced to nanometer range it is very well suited for targeted drug deliveries and so on. Also when the size is in micrometer it is good for cell separation. It has high magnetism of any transition metal oxide [5]. Iron oxide holds relatively a low band gap and good chemical stability.

In this work, iron oxide is prepared from novel sonication method. The aim of this study is to reveal the structural, morphological vibrational and optical

properties of the material. The chemical analysis was performed with XRD (X-Ray Diffraction), FTIR (Fourier Transform Infrared Spectrometer), SEM (Scanning Electron Microscope) and UV-Visible analysis.

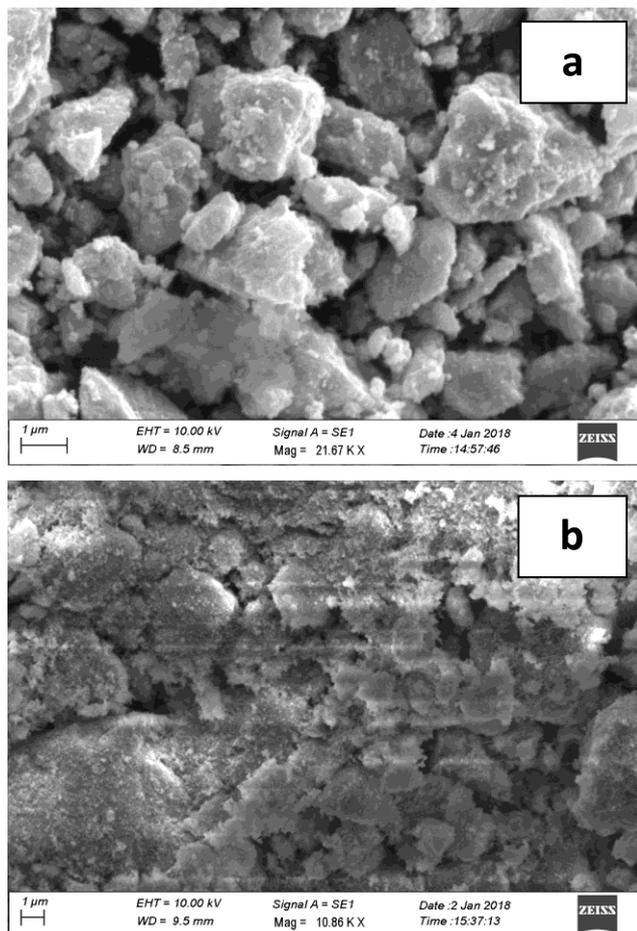
### 2 Experimental

A stoichiometric amount of Ferric nitrate and urea were brought in powder form from Merck to synthesize the pure nano-sized iron oxide particles. 0.1 mol of Ferric nitrate and 0.2 mol of urea were dissolved in 400 ml of double distilled water and stirred with the help of magnetic stirrer for 15 minutes. Ammonia solution was used during the stirring process in order to maintain a pH value of 9. The prepared solution was then transferred to the sonicator and agitated for required hours. The final product was collected and washed several times with double distilled water. The final product was dried at 100 °C for 24 hours. Then, the prepared sample was calcinated at 400 °C for 1 hour using muffle furnace.

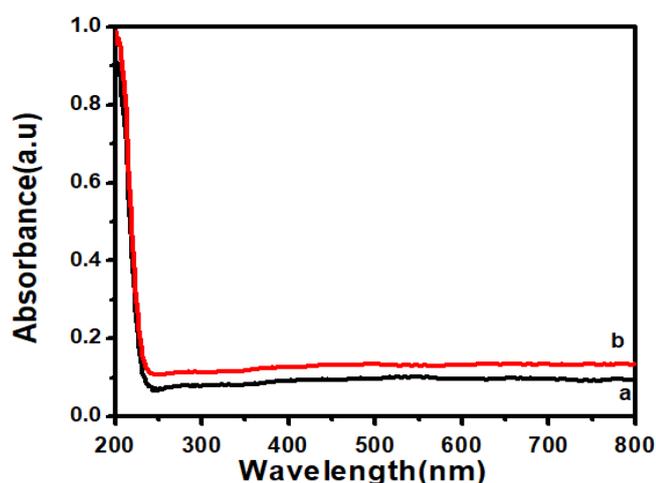
### 3 Results and Discussion

The asprepared and calcinated iron oxide nanoparticles were subjected for various characterizations. As the morphology of the sample plays a vital role in tuning the performance of the sample. The morphology of the prepared nanoparticles was studied using Scanning Electron Microscope model SEM Quanta 200. SEM images of the nanoparticles prepared by sonication method are shown in Fig.1. The SEM micrograph in Fig. 1 (a & b) correspond to the as-prepared sample and sample calcinated for 400°C respectively. From the SEM images, it can be seen that both the samples possess more agglomerations. However on calcinating the sample reduction in agglomeration is witnessed through loosely bound particles here and there.

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**Fig.1.** SEM micrograph of iron oxide nanoparticles (a) as-prepared (b) sample calcinated at 400 °C for 1hours

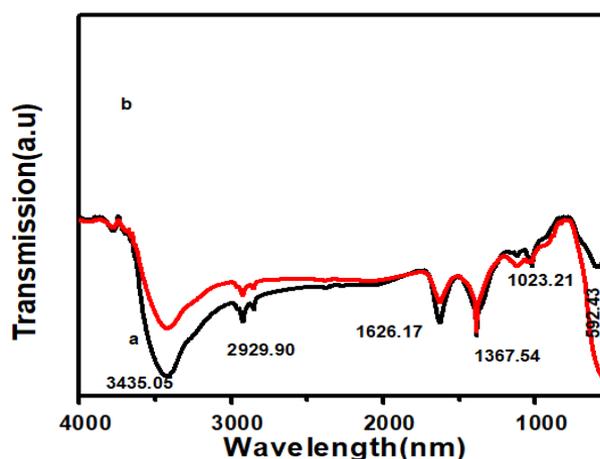


**Fig. 2.** UV-Vis absorption spectrum of (a) as-prepared (b) sample calcinated

The optical property of the synthesized sample was examined by Perkin Elmer make Lambda 35 UV-Vis Spectrometer. The absorbance spectra of the as-prepared sample and the sample calcinated for 400 °C respectively are shown in Fig.2. Both as prepared and calcinated

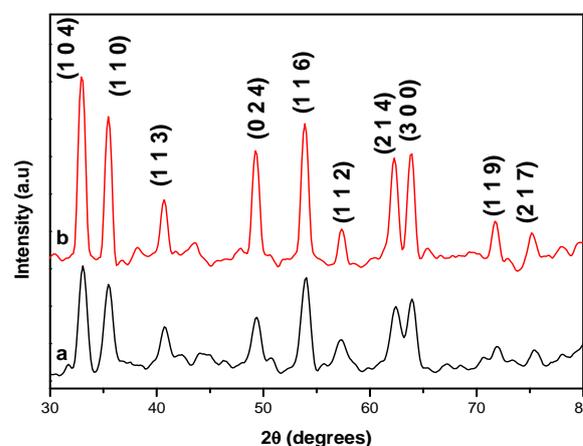
samples show good transparency in the entire visible range. However the calcinated sample shows little increased absorption [6] when compared to the asprepared sample. This may be due to the higher surface to volume ratio of these samples when compared with those samples calcinated at 400 °C.

The Fourier Transform Infrared Spectrometer is used to find out the functional groups present in the sample. The FTIR spectrums of the as-prepared and calcinated samples are shown in Fig. 4. The broad band around 3435  $\text{cm}^{-1}$  and 1626  $\text{cm}^{-1}$  is due to the O-H vibration of the water molecules absorbed to the nanoparticle surface. The strong absorption band at about 592  $\text{cm}^{-1}$  is because of the Fe-O stretching vibration of the Iron Oxide.



**Fig.3.** FTIR Spectra of the prepared sample (a) as-prepared (b) calcinated

The samples prepared were subjected to XRD in order to study the crystallite structure and size. The XRD patterns of the as-prepared and sample calcinated for 1 hour at 400°C are shown in the Fig. 4 (a and b) respectively.



**Fig 4.** XRD Pattern of (a) as-prepared (b) sample calcinated at 400 °C for 1 hours

The XRD pattern of calcinated sample exhibit peaks at  $2\theta=33.14, 35.61$ , which could be indexed to diffraction from the (1 0 4) and (1 1 0) planes and are in good match with JCPDS card # 89-8104 confirming the formation of  $Fe_2O_3$ . However, the intensity of the XRD peaks increase on calcinations which in turn reveals an increase in degree of crystallinity on calcinations [7]. The structure of the iron oxide nanoparticles found as per this XRD data corresponds to rhombohedral structure. The average sizes of the crystallites were determined from the FWHM of the XRD peaks using Debye Scherer equation and were found to be 17.3 nm and 28.85 nm for the as-prepared and calcinated samples respectively.

#### 4 Conclusions

Rhombohedral structured iron oxide nano particles have been successfully synthesized by simple and cost effective sonicator method. FTIR studies reveal the functional group present inside the sample and thereby the presence of iron oxide is obtained. The structural analysis shows the prepared sample is in rhombohedral structure with increasing crystallite size. The absorbance spectrum shows slight rise in absorption and the SEM show less agglomeration when calcinated. Iron oxide is already proven that it has many magnetic properties and therefore is have severe applications.

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#### References

- [1] R. H Khodama, Journal of Magnetism and Magnetic Materials. 200(1999) 359-372.
- [2] R.M. Cornell, U. Schwertmann, second ed. Wiley-VCH, Weinheim. (2003) 48.
- [3] Jongnam Park, Eunwoong Lee, Angew. Chem. 44 (2005) 2872 – 2877.
- [4] Arnaud Demortiere, Daniel Guillon, Nanoscale, 3 (2011) 22-27.
- [5] Jhunu Chatterjee, Yousef Haik, Journal of Magnetism and Magnetic Materials. 257(2003) 113–118.
- [6] Kaushik Gupta, Arjun Maity, Journal of Hazardous Materials, 184(2010) 832-842.
- [7] Paula Soares, Ana Alves Journal of Colloid and Interface Science, 419(2014) 46–51.