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A facile method for the synthesis of magnetite nanocrystals

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Magnetite Nanocrystals (Fe_3O_4) have been synthesized by thermal decomposition synthesis method using $Fe(acac)_3$ and Tri-Octylamine and Oleic acid at a low temperature of 250 ^{0}C compared to other methods. The phase formation for the obtained product was confirmed by powder X-ray diffraction (XRD) and the morphology was analyzed by Transmission Electron Microscopy (TEM). XRD patterns reveal the formation of Magentite (Fe_3O_4) structure in single phase with good crystallinity and TEM pictures shows the formation of mono disperse cubical particles and the size of the particles was found to be around 15 nm which is in accordance with d_{xrd}. The concentration of oleic acid was varied and to study the morphological changes.

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1. Introduction

During the past years, the preparation and characterization of nanoscale materials have an important branch of materials research and attracted considerable attention from both fundamental and applied research due to their unique optical, electrical and other properties [1]. To date, a variety of preparative approaches have been investigated and these are resulted in nanomaterials with controlled size and shape and many reviews are now available. Solution phase syntheses may represent the most promising route to nanoparticles in terms of cost, throughput, and the potential for high volume production. Among them Iron Oxide (ferromagnetic, ferromagnetic and antiferromagnetic materials) is prominent and attracted researchers due to many promising industrial and biomedical application such as catalysis, magnetocooling, optical and recording devices, purification of enzymes and other biological materials, and water purification devices [2-5].

To apply superparamagnetic nanoparticles for biomedical applications, the nanoparticles should be monodisperse to have uniform physical and chemical properties for controlled biodistrubution, bioelimination and contrast effects. The magnetic nanoparticles should also have high magnetic moment and can be so modified that they are capable of binding specifically to a biological entity and able to withstand various physiological conditions. Iron oxide nanoparticles, due to their chemical and magnetic stability and low level of toxicity in biological systems, have been widely tested for their use in biomedicine [6]. However, some well-known material problems need to be solved before these nanoparticles can be utilized for any practical application. The iron oxide nanoparticles used for the tests are often polydisperse with large variation not only in size, but also in shape. Consequently, the physical and chemical properties of these particles are not well controlled and important data on biodistribution/ bioelimination in biological systems, which are essential for in vivo applications, are currently difficult to obtain.

Monodisperse ferrite MFe_2O_4 nanoparticles were recently made by a high-temperature reduction /decomposition reaction of metal acetyacetonate[7-9]. The size of the particles was controlled up to 8 nm from the one step reduction/decomposition reaction. Larger size up to 20 nm was made possible by seed-mediated growth in which small MFe_2O_4 nanoparticles were used as seeds and more MFe_2O_4 was coated over the seeds. By controlling the heating parameters, the reaction further led to the ferrite nanoparticles with cube- or polyhedron- like shapes [9-10]. This reduction/decomposition synthesis is complimentary to others reports in iron oxide nanoparticles syntheses from high temperature decompositions [11-16]. The iron precursors used in this new syntheses are commercially readily available and less toxic than the iron pentacarbonyl, $Fe(CO)_5$, a common precursor used in thermal decomposition reaction.

In this context, the goal of our investigation is to prepare monodisperse nano magnetite with narrow polydispersity and high reproducible magnetic loading, to study their magnetic behavior. Briefly, Magnetite was synthesized by thermal decomposition method. Different experiments were conducted to study the principal parameters influencing the nanoparticle size and size distribution.

2. Experimental

2.1 Chemicals

All the chemicals used in the synthesis are procured from sigma-Aldrich and used in the without further purification.

2.2 Synthesis procedure

The synthesis was carried out using standard procedures and commercially available reagents. In this method, to the tri-n-octylamine, oleic acid is added and stirred for few minutes. To this mixture, Fe(acac)3 is added. These contents were transferred to RB flask and subjected to refluxing at 250^{o}C for 6 hours. After cooling, the black solid residue is washed with ethanol and dried at ambient temperature. The experiment repeated by varying the concentration of Oleic acid. The reaction can be scaled up with out any change in phase and morphology of the samples

2.3 Structural and morphology analysis

The synthesized samples were characterized for their structure and morphology by powder X-ray diffraction (Rigaku 2000 diffractometer) and transmission electron microscopy (JEM-2010, 200kV). The X-ray diffraction data were recorded by using Cu Kα radiation (1.5406 Å). The intensity data were collected over a range of 2θ of 2-60^{o} by counting time of 0.5s per 0.045^{o}. The average crystallite size of the samples was estimated with the help of Scherrer equation using the diffraction intensity of all prominent lines.

3. Results and discussion

3.1. XRD

X-ray diffraction studies confirmed that the synthesized materials were iron oxide of phase magnetite and all the crystal structures agreed with the reported Data. Fig. 1 shows the powder XRD pattern of the sample as a representation of all samples. A definite line broadening of the diffraction pattern is an indication that the synthesized materials are in nanoscale range. The crystalline size was calculated from Scherrer equation (equ. 1) applied to the major peaks and was found to be around 8 nm. The lattice parameters calculated were also in accordance with the reported value.

$$D = \frac{0.94\lambda}{\beta \cos\theta} \longrightarrow (1)$$

Where λ is the X-ray wavelength, θ the Bragg angle for the corresponding plane and β is the pure full width of the characteristic diffracted peak at half of the maximum intensity.

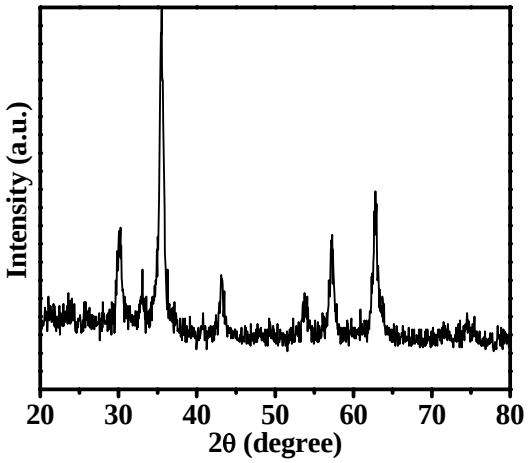

Fig. 1. XRD patterns of synthesized Fe_3O_4 nanoparticles.

3.2. Transmission electron microscopy

Fig. 2 gives the TEM micrographs of the Fe_3O_4 nanoparticles. TEM micrographs reveals that the particles are monodisperse and cubical in structure. The particle size was found to be around 15-20 nm, which was inaccordance with d_{xrd} value.

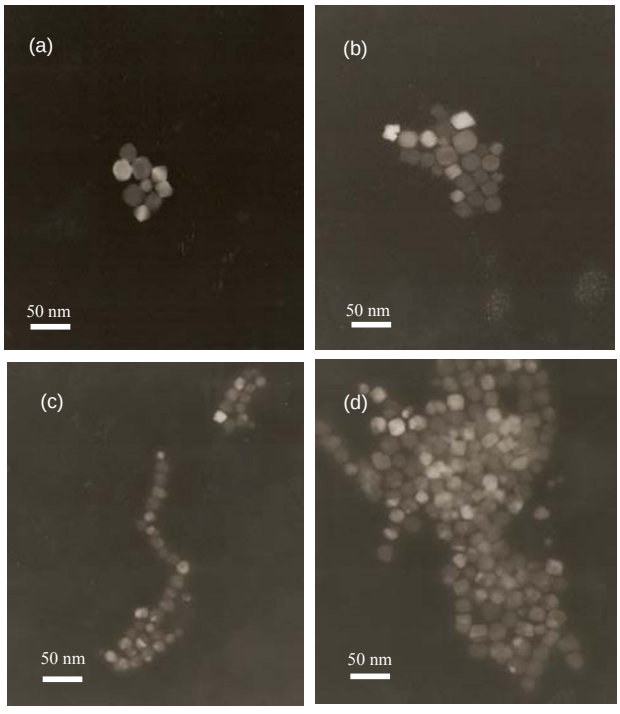

Fig. 2. TEM images of synthesized Fe_3O_4 nanoparticles from a solution of Fe(acac)3 : oleic acid (a) 1:1 (b) 1:2 (c) 1:3 (d) 1:4.

4. Conclusion

The following are the major conclusions from the above study:

1. We synthesized Fe_3O_4 nanoparticles by simple method- decomposition at low temperature compared with other methods.
2. The synthesized particles are monodisperse and cubical.
3. The physical and chemical properties of the synthesized particles are well controlled and applicable for potential application especially like high reproducible magnetic loading and catalysis.

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