Synthesis and Characterization of Magnetite Fine Particles by Simple Co Precipitation Method

Juliya Khanam*, Rownok Jahan, Lutfor Rahman, Sabrina Mostofa, Nahid Sharmin

Abstract— An innovative synthetic method for preparing magnetite (Fe_3O_4) fine nanoparticles was achieved by simple co-precipitation method using ferric and ferrous salt solution in alkaline medium. Magnetite fine nano particles were prepared by mixing and stirring of ferric and ferrous salt at the molar ratio of 2:1 at room temperature. The mixture was treated by adding 25% ammonium hydroxide. The structure morphology and particle size of the synthesized Fe_3O_4 were characterized by X Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and dynamic light scattering (DLS) method. Magnetic property also checked by magnetic bar manual test. The result of XRD characterization was indicated Fe_3O_4 as the product without any contamination. Dynamic light scattering (DLS) of the Fe_3O_4 showed that fine particles of Fe_3O_4 have mean diameter of 350 nm. The main aim of this research was to establish an innovative way to prepare pure and good crystalline magnetic product.

Index Terms— Co Precipitation, DLS, Magnetite, SEM, XRD

1 Introduction

Modern science nanotechnology is one of the most important research sector because these materials represent an intermediate dimension between bulk materials and atoms [1]. By developing the nanotechnology in the last decades, the magnetic nanoparticles have found the special importance in the modern purpose like biomedical sciences caused by their unique characteristics [2], [3], [4]. Nano particles are those structures with at least one dimensions below 100nm which are called ultrafine nanoparticles and a second, dimension below 1µm are known as fine nanoparticles [5]. Among different nanoparticles recently Fe_3O_4 magnetic fine nanoparticles have been intensively investigated because of their variety of scientific and technological applications, such as biosensor [6], antimicrobial activity [7], food preservation [8], removal of dyes from waste water [9], magnetic storage media, cell sorting and targeted drug delivery [10, [11],12]. Besides, it has also been widely used in biomedical research because of its biocompatibility and magnetic properties [13]. In this work, an innovative invention was done which relates to a process of preparing magnetic fine nanoparticles by the simple co-precipitation method [14]. Co-precipitation method for synthesis of Fe_3O_4 which is easy to do with the success rate from 96 to 99.9% [15]. The modified method of synthesis Fe_3O_4 magnetic fine nanoparticles with narrow size distribution and excellent dispersion in fluids [16]. The main objective of this research work was to optimize the co-precipitation method for the preparation of magnetic without any contamination of other iron oxides and also to study the effects of the synthesis parameter such as stirring rate and reaction temperature on the physical properties of the magnetic particles.

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2 Materials And Methods

2.1 Materials and characterization

All the reagents used for the synthesis of Fe_3O_4 were analytical grade. Moreover they were used in pure form and purchased (Merck, Germany). All the chemicals were used without furt her treatment. Hydrated Ferrous sulfate, Hydrated ferric choloride, Ammonium hydrooxide, ethanol were used in this work.

The phase composition of the synthesized magnetite was determined by X-ray diffraction analysis by an EMMA, GBC Corporation, Australia diffractometer. The diffraction pattern was recorded using CuKα radiation (λ=1.5406 A^0) in the range 10-80 degrees with scanning speed 2degree/minute. Phase analysis of magnetite was confirmed by comparing the d values and intensity ratios of the diffraction lines in the recorded patterns with standard data of the PDF file 00-02-1035. Scanning electron microscope (EVO 18, Curl Zeiss AG, Germany) was used to observe the morphology and mean particle size. Particle size distribution was observed through dynamic light scattering method (DLS) using Zetasizer Nano ZS90 (ZEN3690, Malvern Instruments Ltd., UK).

2.2 Synthesis of magnetite fine particles

Magnetic fine nanoparticles were synthesized by simple co-precipitation method. The synthesis was carried out by co-precipitation of ferric/ferrous salt in the molar ratio of 2:1 in aqueous solution with alkali along with suitable aging at room temperature. Briefly 10.82g $FeCl_3.6H_2O$ and 5.55g $FeSO_4.7H_2O$ were dissolved in 200ml deonized water and stirred well for 15 min at 250-300 rpm for complete dissociation. After stirring chemical precipitation was achieved at room temperature by adding 25% NH_4OH with constant stirring, until the p^H be-

tween 8 to14 for complete precipitation. The obtained precipitate were heated at 80^0 C for 30 minutes in a round bottom flask with stirring, a black precipitate was obtained exhibiting strong magnetic property. The precipitates were filltered and separated by a strong magnet and washed with hot deionized water and ethanol. The synthesized magnetite was dried in an oven at 70^0 C for two days and it was finally grinded and sieved. The chemical reaction of formation may be shown in equation (1).

$$2Fe^{3+} + Fe^{2+} + 8OH^- \longrightarrow Fe_3O_4 + 4H_2O \text{ …….. (1)}$$

2 Result and Discussion

The crystallinity of the magnetite sample was investigated by XRD as shown in Fig 1. The X-ray powder diffraction (XRD) patterns of fine particles confirmed that the synthesized product was pure magnetite. The results show that the sample has six peaks at 2θ/degree of 30.32°, 35.78°, 43.38°, 53.82°, 57.30° and 62.90° representing the corresponding indices of (220), (311), (400), (422), (511) and (440) respectively of a pure Fe_3O_4 with a spinal structure (file PDF no-00-002-1035). The peak indicates that Fe_3O_4 with a spinal structure and no characteristics peak of impurities are detected in the XRD patterns.

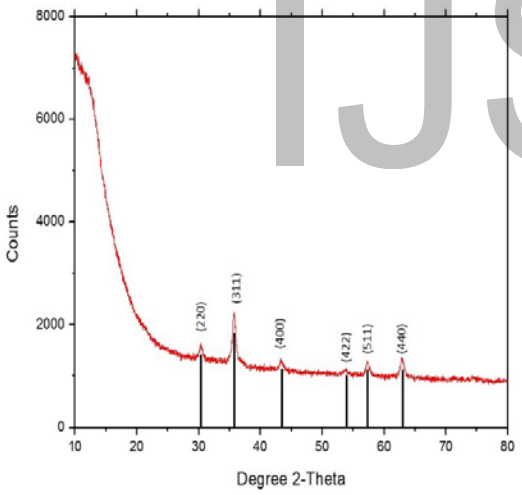

Fig 1: XRD pattern of magnetite fine particles

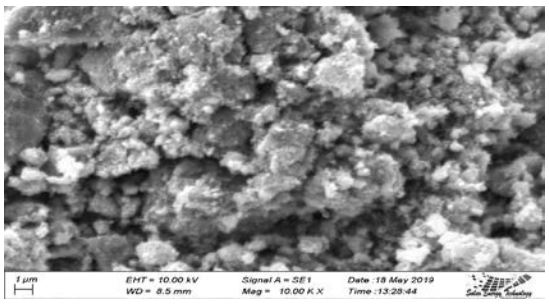

Fig 2: SEM image of magnetite fine particles

It is evident from the picture that the particles are in a nano scale and composed of small particles. From the image it confirms that the Fe_3O_4 fine particles are cubic and highly uniform in size.

Dynamic light scattering (DLS) method was used to measure particle size of the Fe_3O_4. From Fig 3, it is observed that the system was homogeneous, coagulation and impurity not present and the particles range approximately between 250 nm to 480 nm with mean particle size of 350 nm which indicates that magnetite composed of fine nanoparticles.

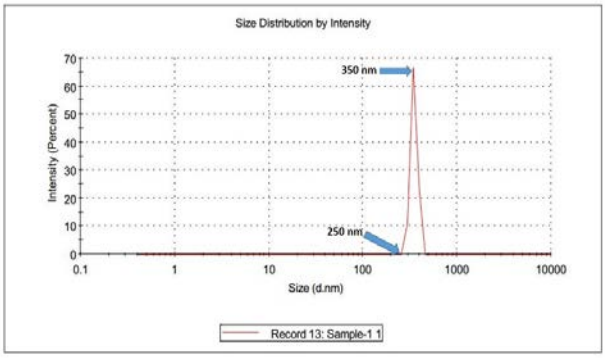

Fig 3: DLS particle size analysis curve of magnetite fine particles

Magnetic property of the synthesized product was initially checked with a magnetic bar. All the product has magnetic property, some are strong magnetic while some are less (Fig 4)

Fig 4: Magnetic property measurement by nagnetic bar

6 Conclusion

The present investigation summarized the preparation and characterization of magnetite fine particles by simple co-precipitation method. The XRD result showed that the synthesized fine magnetic particles are well crystallized and can be indexed into spinal structure without any contamination. DLS method determined the average particle size of magnetite is 350 nm. The morphological observations indicated that the

particles are cubic and uniform in size. The practical approach of magnetite production could be a potential, cost effective and eco-friendly method. The prepared fine magnetite particles could be use in different application due to its magnetic property.

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