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Synthesis and Characterization of Superparamagnetic Magnetite Nanoparticles for Drug Delivery Application

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ABSTRACT

Magnetic nanoparticles have been extensively studied because of their potential applications as contrast agents in Magnetic Resonance Imaging (MRI) of tumors, cell and DNA separation, magnetically guided drug delivery, tumor hyperthermia. Among the magnetic oxides, magnetite nanoparticles are most suitable due to their low toxicity and good magnetic properties which may be used in drug delivery. Magnetite nanoparticles were synthesized using $FeCl_3$ and $FeSO_4$ as precursors and characterized for size and shape using non-contact Atomic Force Microscopy (nc-AFM). The formation of magnetite was confirmed by XRD pattern. The elemental composition of the obtained phase was determined using Energy Dispersive Analysis of X Rays (EDAX). In this work, we are aiming to develop drug loaded biopolymer Magnetite nanoparticles for biomedical application. Our main objective is to synthesize and characterize Magnetite (Fe_3O_4) nanoparticles.

Keywords: Drug delivery, Magnetite, Nanoparticles

INTRODUCTION

Magnetic nanoparticles have been extensively studied because of their potential applications as contrast agents in Magnetic Resonance Imaging (MRI) of tumors, cell and DNA separation, magnetically guided drug delivery, tumor hyperthermia etc. [1]. Among the magnetic oxides, magnetite nanoparticles are most suitable due to their low toxicity and good magnetic properties. The small size customized surface, improved solubility and multi-functionality of nanoparticles will continue to open many doors and create new biomedical applications. Indeed, the novel properties of nanoparticles offer the ability to interact with complex cellular functions in new ways. This rapidly growing field requires cross-disciplinary research and provides opportunities to design and develop multifunctional devices that can target, diagnose, and treat devastating diseases such as cancer. In this work, we are aiming to develop drug loaded biopolymer Magnetite nanoparticles for biomedical application. Magnetic nanoparticles offer some attractive possibilities in biomedicine. They have controllable sizes ranging from few nanometers up to 10 nanometers, which places them at dimensions that are smaller then or comparable to those of cell (10-100 μm), a virus (20-450 nm), protein (5-50 nm). This means that they can get close to a biological entity of interest. Indeed, they can be coated with biological molecules to make them interact with or bind to a biological entity, thereby providing a controllable means of tagging or addressing it. The nanoparticles are magnetic, which means they obey Coulomb's law and can be manipulated by an external magnetic field gradient [2-5]. Our main objective is to synthesize and characterize Magnetite (Fe_3O_4) nanoparticles which may be used in drug delivery and this will be dealt with in a separate communication.

MATERIALS AND METHODS

Synthesis of magnetite nanoparticles

Method A: A solution mixture of 0.1 M KOH and 0.05 M $FeSO_4$ were mixed in a three neck round bottomed flask under N_2 bubbling. Then 0.2 M of KNO_3 was dropped in to the solution. The temperature was maintained at 90°C by using an oil bath setup. The solution was heated for 24 h along with stirring. Finally, a black precipitate was formed. The formed black precipitate was washed with ethanol and deionized water. Then, the magnetite nanoparticles were dispersed in ethanol.

Method B: A solution mixture of $FeCl_3$ (0.085 M) and $FeSO_4$ (0.05 M) at pH 1.98 were mixed in a three neck round bottomed flask under N_2 bubbling. In the process, nitrogen was used to keep the system under the atmosphere of non-oxygen so as to protect iron salts from oxidation [6]. Then, ammonia aqueous solution (1.5 M) was dropped in to it and violently stirred with the help of magnetic stirrer until the pH of the solution rise to 9. Immediately a black precipitate appeared showing the formation of magnetite nanoparticles. pH value was noted simultaneously with the help of pH paper. The obtained magnetite nanoparticles were washed immediately with water and ethanol. Finally, magnetite nanoparticles dispersed in ethanol.

RESULTS AND DISCUSSION

Characterization of magnetite nanoparticles using Atomic Force Microscopy (AFM)

The size and size-distribution of magnetite nanoparticles were investigated by AFM. AFM images were optimized at different imaging parameters like set point, scan rate and feed-back gain (Typical values-0.05080, 1.00 lps and 0.1). The instrument was operated in Non-Contact Mode. The samples for AFM were prepared by depositing one drop of magnetite nanoparticle solution on top of a cleaved flat mica substrate. AFM images are shown in Figure 1 for two different regions on the sample. The images show that the particles are uniformly spherical in shape and the diameters of the particles were found to be in the range 5-50 nm. The average particle size is around 10 nm as shown in the histogram in Figure 1.

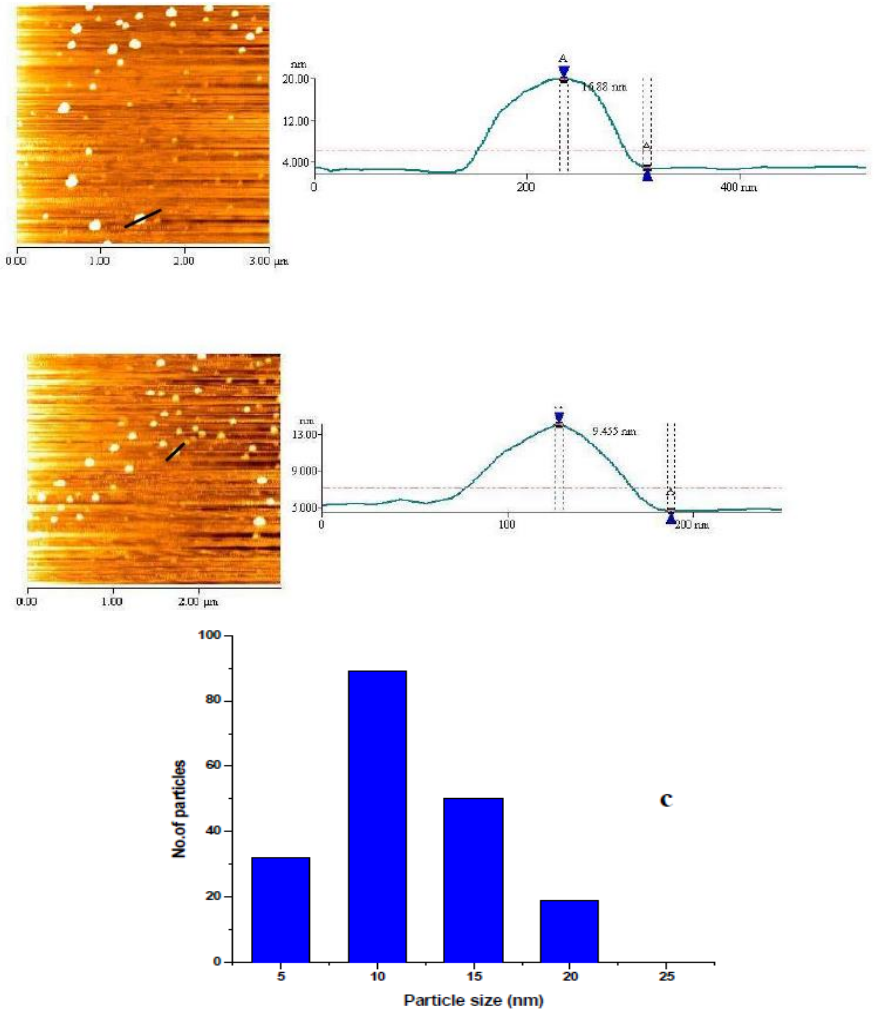

Figure 1: Non-contact mode AFM image of scan size 3 µm, showing spherical magnetite nanoparticles and (c) Histogram of magnetite particle size distribution

Scanning Electron Microscopy (SEM)

SEM was done to determine the morphology of the magnetite nanoparticles and the results are shown in Figure 2. It can be seen from the figure that the resulting magnetite nanoparticles are almost spherical and quite small. The approximate particle size is around 13 nm.

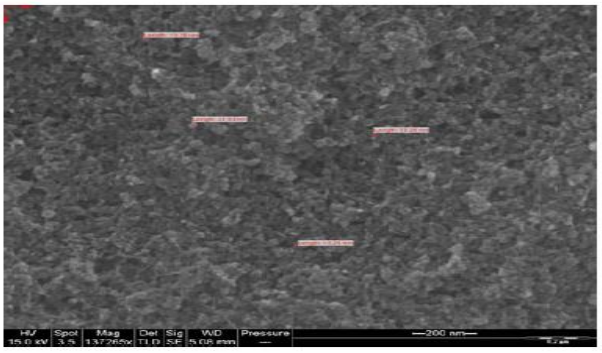

Figure 2: SEM morphology of magnetite nanoparticles

X-Ray Diffraction (XRD)

X-Ray diffraction is a useful technique used to determine the crystal structure of a material. XRD was done on Fe_3O_4 nanoparticles to confirm that the obtained phase is magnetite (Fe_3O_4) and not a different iron oxide or its derivative. Polymer coated magnetite nanoparticles were not characterized with XRD because the polymer is not crystalline so it is not expected to contribute anything to diffraction. The position of all diffraction peaks matched with standard Fe_3O_4 diffraction data. Figure 3 shows the X-ray diffraction patterns of the formed Fe_3O_4 nanoparticles.

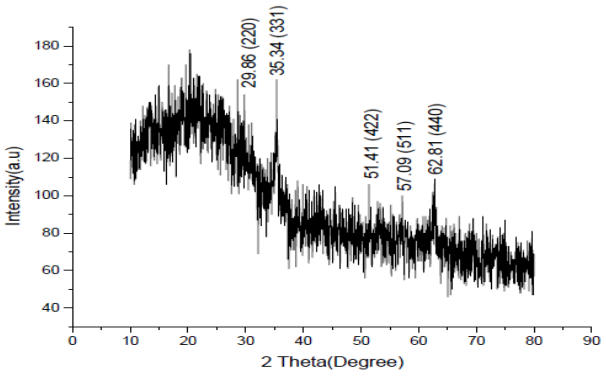

Figure 3 X-Ray diffraction patterns of the Fe_3O_4 nanoparticles

Energy Dispersive Analysis of X Rays (EDAX)

The EDAX spectrum of the synthesized Fe_3O_4 nanoparticles, presented in Figure 4, was performed to confirm the elemental composition of the formed product. The ratio of the element Atomic percentage was found to be 43.85/56.15=0.78. This value is quite close to that of the expected viz. 0.75. It is clear from this, that the nanoparticles consist of Fe and O elements in the appropriate ratio, further confirming the formation appearance of Fe_3O_4 nanoparticles.

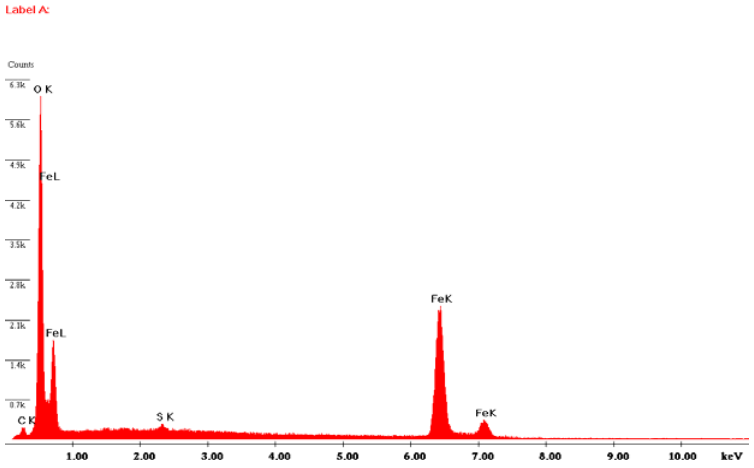

Figure 4: The EDAX spectrum of synthesized Fe_3O_4 nanoparticles

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR of magnetite nanoparticles is shown in Figure 5. The spectra shows peak around 572 cm^{-1}, which is typical characteristic of Fe-O-Fe in Fe_3O_4 sample as reported in literature [7].

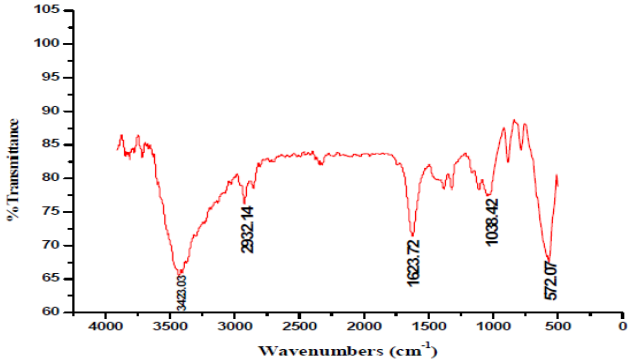

Figure 5: FTIR spectrum of magnetite nanoparticles

CONCLUSION

Magnetite nanoparticles were synthesized using $FeCl_3$ and $FeSO_4$ as precursors. The magnetite nanoparticles were characterized for size and shape using non-contact AFM. Synthesized Fe_3O_4 nanoparticles were spherical in shape and the height of the particles was in the range of 5-50 nm with average particle size of 10 nm. The formation of magnetite was confirmed by XRD pattern. The elemental composition of the obtained phase was determined by EDAX, with the ratio of Atomic % found to be ~0.78, which is close to the expected value of 0.75.

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