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SYNTHESIS AND CHARACTERIZATION OF IRON COBALT (FECO) NANORODS PREPARED BY SIMPLE CO-PRECIPITATION METHOD

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ABSTRACT

We report here a simple synthesis method for iron-cobalt (FeCo) nanoparticles by iron nitrate ($FeNO_3.9H_2O$) and cobalt nitrate ($CoN_2O_6.6H_2O$) as precursor in the presence of ethanol agent and cetyltrimethylammonium bromide (CTAB) surfactant. The samples were characterized by high resolution transmission electron microscopy (HRTEM), field effect scanning electron microscopy (FESEM), X-ray diffraction (XRD) and electron dispersive spectroscopy (EDS) in different temperature. XRD pattern of FeCo samples showed the structure of body center cubic (bcc) structure. The SEM images show that average particle size of as-prepared sample was around 36 nm and annealed samples were around 28 at 800 ^oC. These images also showed the particles changed from rod-liked shaped to sphere-liked shaped by increasing annealing temperature from room temperature to 800 ^oC. The TEM studies show the rod-liked shaped particles. The sharp peaks in FTIR spectrum determined the element of Fe-Co nanoparticles.

Keywords: FeCo nanoparticles, sodium borohydrid, CTAB, chemical synthesis

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1. INTRODUCTION

Magnetic nanoparticles with tailored surface modifications have been widely used experimentally for numerous in vivo applications such as hyperthermia, medical diagnostics, therapeutics, and drug delivery [1–5]. FeCo alloys are important soft magnetic materials because of their unique magnetic properties including high permeability, very high saturation magnetization and high Curie temperature. Recently, FeCo nanoparticles have attracted great interest because of their potential applications in advanced materials and devices such as the building blocks of exchange coupled nanocomposite magnets, ultrahigh density recording media, microwave devices and biomedicines [6–9]. Several chemical and physical methods for the synthesis of FeCo nanoparticles have been reported recently [10–17]. An important aspect in the application of nanoparticles is their stability in ambient conditions. Most chemically synthesized FeCo nanoparticles take a disordered body-centered cubic (bcc) structure and are not stable in air. Heat treatment is generally applied to make these particles air stable [6,8]. As reported, the synthesis path of ferrite nanoparticles is crucial in controlling their composition, shape, size and magnetic properties [18, 19] because other phases, in particular hydroxides, oxyhydroxides, or oxides, can be formed depending on the reaction conditions [20]. Several approaches have been reported to date for the synthesis of Co ferrite species in a variety of shapes and sizes. These include sol-gel [21,22], freeze drying [23], reverse micelle [24], co-precipitation [25, 26], template-assisted [27], solvothermal [28], hydrothermal [29], Langmuir-Blodgett [30] and thermal destruction [31]. In this paper, FeCo magnetic nanoparticles were synthesized using iron nitrate precursor and cobalt nitrate in the presence of CTAB surfactant and ethanol agent using co-precipitation methods. Structural and surface morphological properties are discussed by XRD, HRTEM, FESEM and FTIR analyses.

2. EXPERIMENTAL DETAIL

All materials were purchased from Merck Company. Iron cobalt nanoparticles were synthesized by a simple sol-gel synthesis according to the following manner. Firstly, 10 g iron nitrate ($FeNO_3.9H_2O$) was completely dissolved in 50 mL pure water with stirring at room temperature. In the separate bottle, 10 g, cobalt nitrate ($CoN_2O_6.6H_2O$) was completely dissolved in 50 mL pure water with stirring at room temperature. When two solution was completely dissolved they mixed them to each other and 50 mL ethanol was slowly added to the solution. After that 1 g CTAB surfactant were added to the solution and the mixed

solution stirred with a magnetic stirrer at 85 ^{o}C. The color of solution changed to dark red by adding cobalt sulfate. The pH=5 was maintained during the synthesis. The product were evaporated for 4 hours, cooled to room temperature and finally calcined at 800 ^{o}C for 3 hours. All analyses were done for samples without any washing and purification.

The specification of the size, structure and optical properties of the as-synthesis and annealed nanoparticles were carried out. X-ray diffractometer (XRD) was used to identify the crystalline phase and to estimate the crystalline size. The XRD pattern were recorded with 2θ in the range of 4-85^{o} with type X-Pert Pro MPD, Cu-K_{α}: λ = 1.54 Å. The morphology was characterized by field emission scanning electron microscopy (SEM) with type KYKY-EM3200, 25 kV and transmission electron microscopy (TEM) with type Zeiss EM-900, 80 kV. Fourier transforms infrared spectroscopy (FTIR) with WQF 510.

3. RESULT AND DISCUSSION

XRD analysis was used to identify crystalline phases and to estimate the crystalline sizes. Figures 1 shows the XRD patterns of Iron cobalt after annealing at 800 ^{o}C. The exhibited peaks correspond the body center cubic structure. The mean size of the ordered FeCo nanoparticles has been estimated from full width at half maximum (FWHM) and Debye-Sherrer formula according to equation the following:

$$D = \frac{0.89\,a}{B\cos} \qquad (1)$$

where, 0.89 is the shape factor, λ is the x-ray wavelength, B is the line broadening at half the maximum intensity (FWHM) in radians, and θ is the Bragg angle. The mean size of as-prepared samples was around 36 nm from this Debye-Sherrer equation.

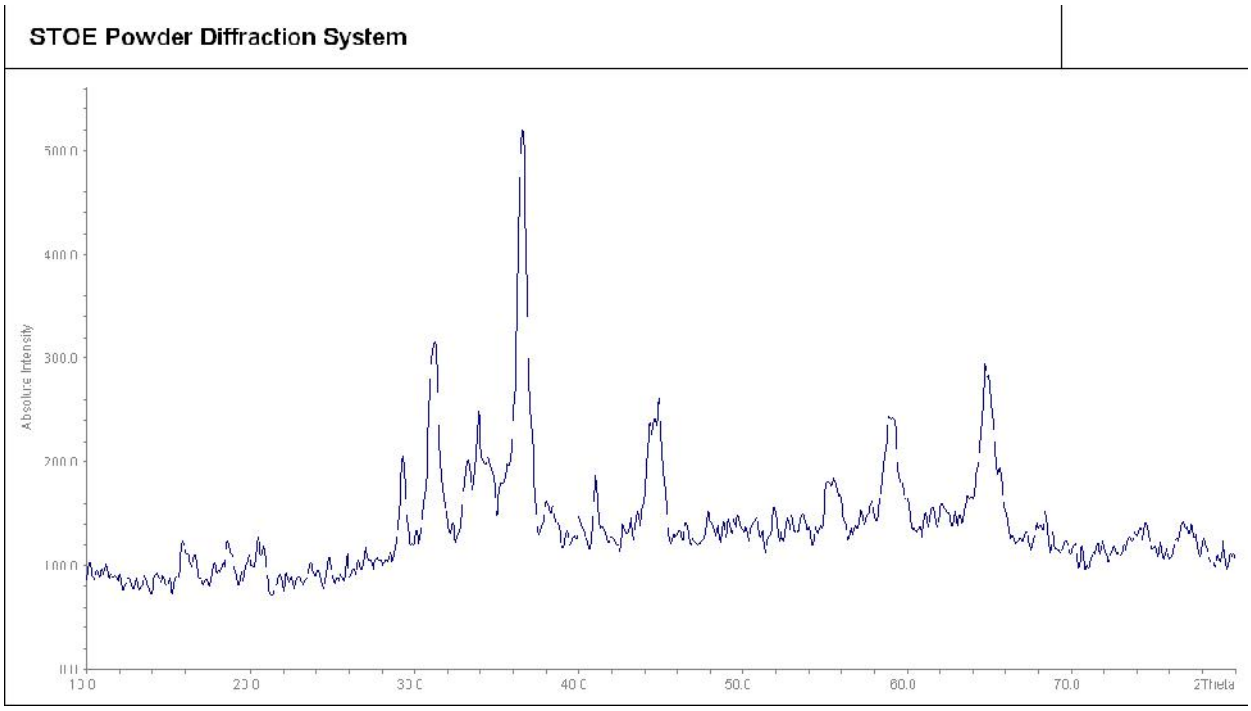

Fig.1. XRD patterns of annealed FeCo samples

SEM analysis was used for the morphological study of nanoparticles of samples. These analyses show that high crystallinity emerged in the samples surface by increasing annealing temperature. With increasing temperature the morphology of the particles changes to the sphere-like shaped and nanopowders were less agglomerate. Figure 2(a) shows the SEM image of the as-prepared FeCo nanoparticles ith average partile size of 36 nm prepared by this method. In this figure, the particles are rod-liked shaped with formation of clusters. Figure 2(b) shows the SEM image of the annealed FeCo nanoparticles at 800^{o}C for 3 hours with average particles size is 28 nm. The particles change from rod-like shaped to sphere-liked shaped by increasing annealing temperature.

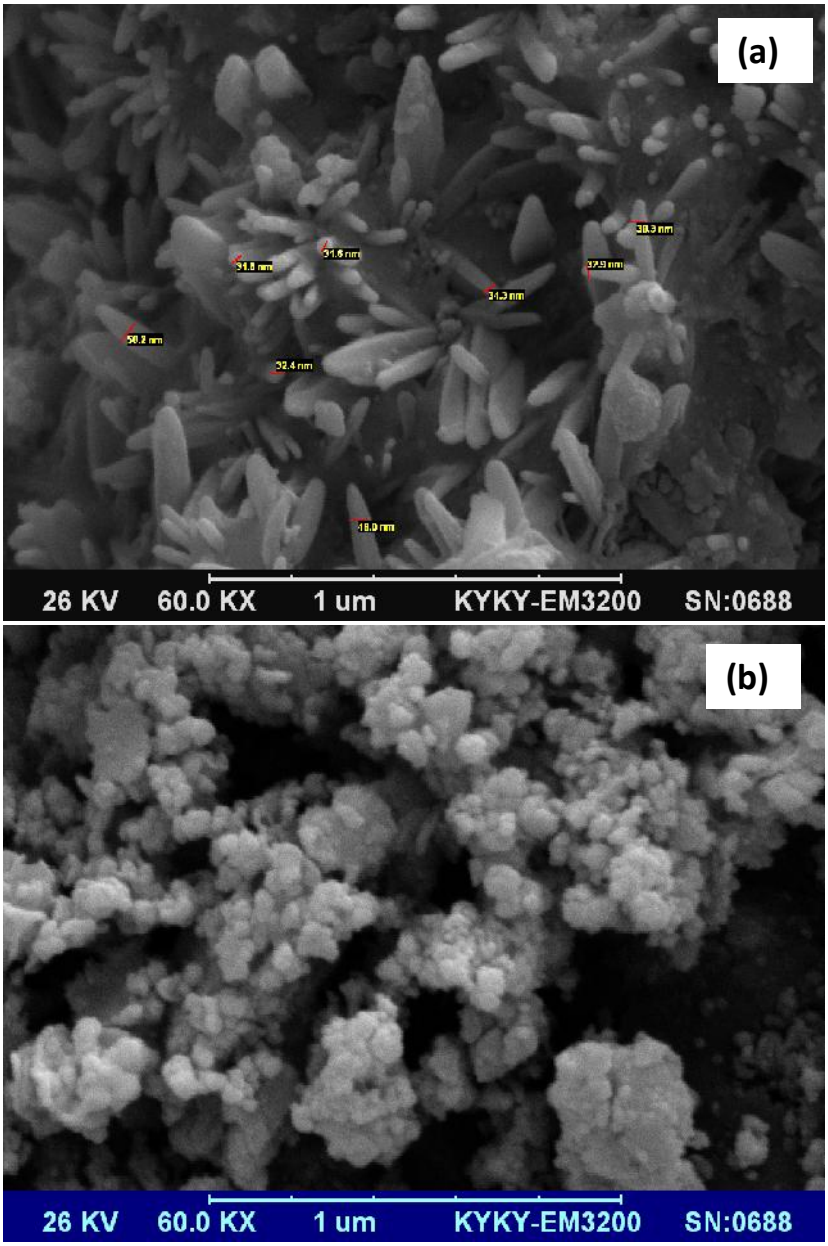

Fig.2. SEM images of the (a) as-prepared (b) annealed FeCo nanoparticles at 800^0C.

The transmission electron microscopic (TEM) analysis was carried out to confirm the actual size of the particles, their growth pattern and the distribution of the crystallites. Figure 3 shows the as-synthesized TEM image of rod-like FeCo nanoparticles with diameter of 36 nm prepared by chemical synthesis route. It can be seen that rod particles were prepared with less aggregation.

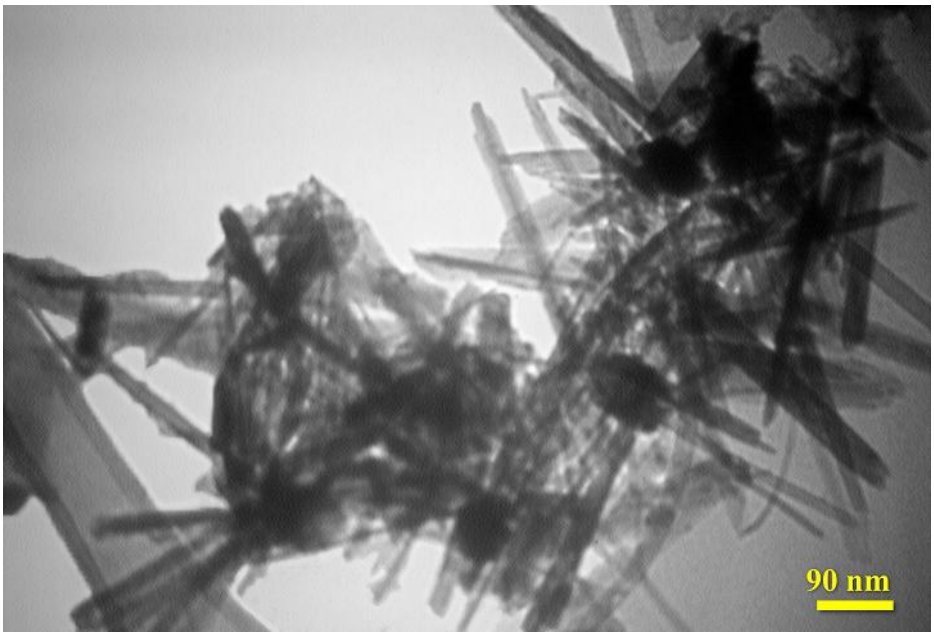

Fig.3. TEM image of the as-prepared FeCo nanoparticles

Figure 4 shows the size measurement of 100 randomly selected particles. By fitting it with a log normal curve leads to a measured mean diameter of 36 nm with standard deviation of about 10%

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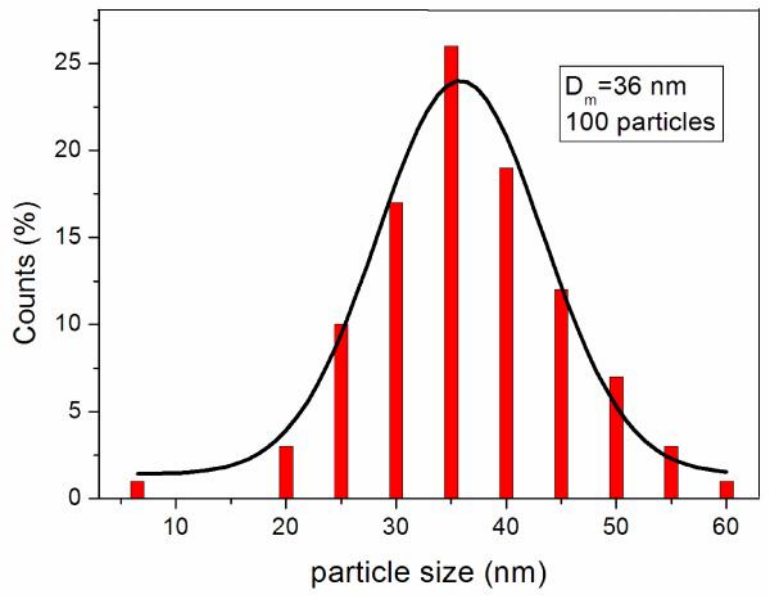

Fig.4. Particle diameter histogram of as-prepared FeCo nanoparticles

In figure 5, the infrared spectrum (FTIR) of the annealed FeCo nanoparticles was in the range of 400-4000 cm^{-1} wavenumber which identify the chemical bonds as well as functional groups in the compound. The large broad band at 3398 cm^{-1} is ascribed to the O-H stretching vibration in OH^- groups. The absorption peaks around 1639 cm^{-1}, 1489 cm^{-1} are due to the

asymmetric and symmetric bending vibration of C=O. The strong band below 700 cm^{-1} is assigned Fe-O stretching mode. The bands corresponding to Fe-Co stretching mode are seen at 661 cm^{-1}, 609 cm^{-1} and 583 cm^{-1}.

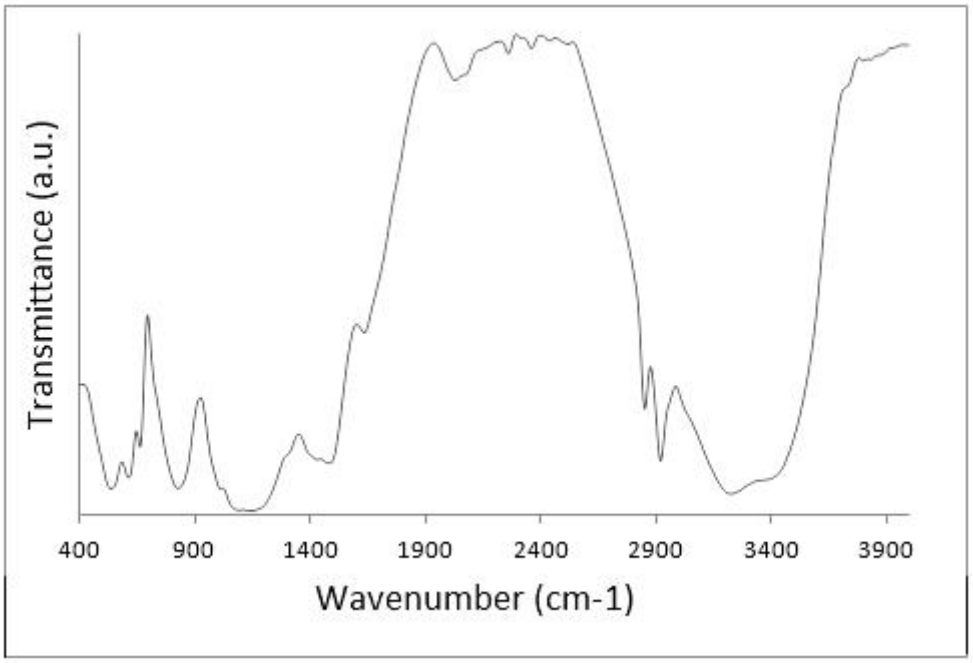

Fig.5. EDS spectra of the annealed FeCo particles

4. CONCLUSION

The Fe-Co nanoparticles have been successfully synthesized using iron nitrate and cobalt nitrate in the presence of ethanol agent and CTAB surfactant. XRD spectrum shows body center cubic structure of the samples. From SEM images, it is clear that with increasing temperature the morphology of the particles changes to sphere-like shaped with less agglomeration. TEM image exhibits that the as-synthesized FeCo nanoparticles with an average diameter about 36 nm and annealed one about 28 nm at 800 ^{o}C. From the FTIR data, it is shown the presence of Fe-Co stretching mode.

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