

## Facile synthesis of high magnetization long term stable bimetallic FeCo nanoparticles

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### Abstract

In this study, we reported a facile synthesis of bimetallic FeCo nanoparticles (Fe-Co NPs) by  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  and  $\text{Co}(\text{Ac})_2 \cdot 4\text{H}_2\text{O}$  in the presence of sodium borohydride and 2-thiolic acid. The structure and morphology of the nanoparticles were characterized by X-Ray Diffraction (XRD), Energy-dispersive X-ray spectroscopy (EDS), and Transmission Electron Microscopy (TEM). These small, spherical shape and pure phase FeCo bimetallic nanoparticles have saturation magnetization up to 221 emu/g. The results suggest that the long term stable and high saturation magnetization ( $M_s$ ) FeCo nanoparticles can be used for data storage, catalysis, environmental remediation, high-performance inductors, magnetic hyperthermia treatment and magnetic resonance imaging applications.

**Keywords:** Bimetallic Nanoparticles; Facile Synthesis; FeCo Nanoparticles; High Saturation Magnetization; Magnetic Property.

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### INTRODUCTION

FeCo nanoparticles have attracted a lot of attention because of their excellent magnetic properties, which make them promising candidates for various technical and industrial applications such as data storage, microwave absorption, electro-catalytic activity, high-performance inductors, magnetic resonance imaging, biotechnology, and drug delivery [1-5]. The magnetic properties of nanoparticles were found to be strongly dependent on their composition, shape, and size. In this regard, it is important to control the synthesis reaction to obtain more morphologies and wide size distributions. [5, 6].

There are many reports on the synthesis of FeCo nanoparticles, such as mechanical attrition or high-energy ball milling, thermal decomposition, electrochemical, radiofrequency plasma torch, and chemical vapor deposition [7-10]. However, there are some limitations including complex synthesis process, long synthesis duration, high production cost. A chemical way is an ideal

approach to obtain FeCo nanoparticles at a low cost. It can easily control the composition, size, shape, and other parameters that play a vital role in practical applications [11]. Sudfeld *et al.* reported that the saturation magnetization for 12 nm  $\text{Fe}_{65}\text{Co}_{35}$  particles is about 158 emu/g. In addition, the authors report that the particles undergo easy oxidation by air [12]. Chaubey *et al.* reported the synthesis of spherical FeCo nanoparticles with a diameter below 20 nm by reducing  $\text{Fe}^{3+}$  and  $\text{Co}^{2+}$  with 1,2-hexadecanediol at 300 °C. The saturation magnetization of the synthesized particles is size dependent. For the 10 and 20 nm particles, the saturation magnetization at room temperature is about 130 and 210 emu / g, respectively. [13]. Mehdi Zamanpour and his colleague demonstrated that the synthesis of FeCo nanoparticles of pure phase in an average diameter of 30 nm with an saturation magnetization at the optimized ambient temperature of 221 emu/g [14]. F. J. Yang showed that the FeCo nanoparticles were prepared by a polyol reduction method, and the largest  $M_s$  273 emu/g and the lowest

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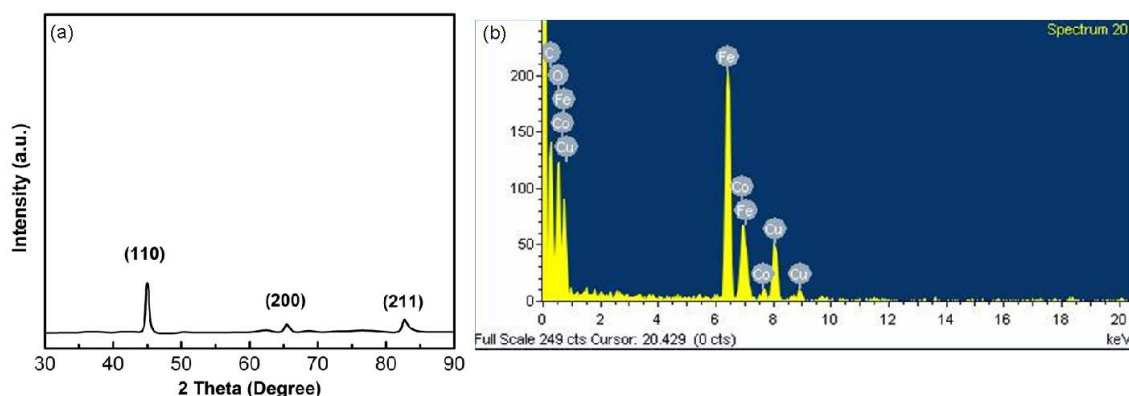


Fig. 1: (a) X-ray diffraction pattern for FeCo nanoparticles, (b) EDX spectrum of as-prepared Fe-Co NPs.

$H_c$  82 Oe were obtained in  $Fe_{55}Co_{45}$  and  $Fe_{48}Co_{52}$  nanoparticles, respectively, at an annealing temperature of 850 °C [15]. Kim et al. also reported the preparation of 20-nm-diameter particles that have a saturation magnetization of 206 emu/g, reducing Fe and Co salts in an aqueous solution [16]. Desvaux *et al.* demonstrated that FeCo nanoparticles synthesized by co-decomposition of organometallic precursors under hydrogen. Superlattice multilayers from FeCo nanoparticles have been successfully obtained [17]. In this work, FeCo bimetallic nanoparticles were synthesized using sodium borohydride as the reductant and 2-thioctic acid as a stabilizer, and the stable, monodisperse bimetallic FeCo nanoparticles show satisfactory microstructure and high Ms.

## EXPERIMENT

### Synthesis of Fe-Co Nanoparticles

In this experiment, 50 mL of water was added in a mixed aqueous solutions of iron sulphate ( $FeSO_4 \cdot 7H_2O$ , 5 gm, 99.00%), cobalt acetate ( $Co(Ac)_2 \cdot 4H_2O$ , 5 gm, 99.00%) and 2-thioctic acid (1 ml). Freshly prepared  $NaBH_4$  (1 ml) was injected quickly into the stirring system. Sodium hydroxide (NaOH, 98%) pellets were subsequently added to maintain the PH. The heater was turned off immediately after the solution turned grey, indicating formation of nanoparticles. The reaction mixture was then allowed to cool to room temperature while maintaining continuous stirring. The prepared nanoparticles were subsequently extracted from the reaction mixture by centrifugation, followed by multiple water and ethanol washes. The precipitates were dried in

vacuum at room temperature for 24 h. After then the collected sample was annealed at 400 °C for 4 h, under an argon atmosphere.

### Characterization

The structural characterization was performed using X-Ray Diffractometer (XRD) (Rigaku Ultima IV) with a Cu target. The particle morphology and size distribution were studied using a transmission electron microscope (TEM) (Tecnai G2 S-TWIN). The compositional and elemental analysis was performed using an energy dispersive X-ray spectrometer (EDS), which is attached to the FE-SEM. Hysteresis measurements at room temperature were performed using a vibrating magnetometer (VSM, LakeShore USA) having the range  $1 \times 10^{-7}$  emu to  $1 \times 10^3$  emu.

## RESULTS AND DISCUSSION

Fig. 1(a), shows the powder x-ray diffraction (XRD) pattern of FeCo bimetallic magnetic nanoparticles. The peaks can be easily attributed to the reflections (110), (200), and (211) of the bcc FeCo nanoparticles (JCPDS No. 49-1568). The crystalline size according to the Scherrer equation is about ~7 nm. It is also worth mentioning that there are no peaks of XRD peaks that correspond to iron or cobalt oxides. The EDS pattern of the synthesized FeCo nanoparticles is also represented in Fig. 1(b), where the EDS peaks correspond to the FeCo nanoparticles and the background peaks correspond to the ligand chemicals present in the coating of the nanoparticles. The proportion of Fe and Co peaks in the EDS also corresponds to the molecular ration of the precursor (1/1) used in the

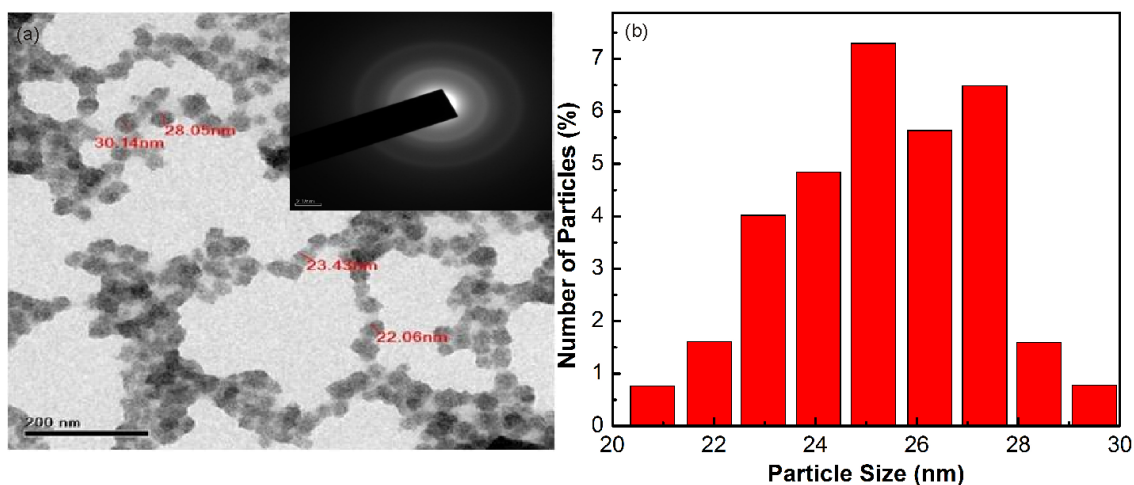


Fig. 2: (a) The TEM image of bimetallic FeCo NPs; inset shows the SAED patterns of as-prepared nanoparticles, (b) Histogram of FeCo bimetallic nanoparticles.

synthesis procedure. In addition, the amount of oxygen due to the formation of oxides was quite low. The Copper peaks visible here are due to the copper grid in the EDX apparatus. The EDX analysis of the preparations supports the presence of FeCo bimetallic nanoparticles in the matrix.

Fig. 2(a) depicts that the typical TEM image of bimetallic nanoparticles prepared with the same molar ratio as iron to cobalt precursors. Judging by the surface plot profile, the FeCo bimetallic nanoparticles appeared as a narrow size distribution and found to be linked with each other in a chain-like morphology. The size distribution of these nanoparticles is homogeneous in nature and spherical in shape. In Fig. 2(b), the histogram shows that the particle size of these nanoparticles is in the range of 20-30 nm. In the upper right region of Fig. 2(a), the selected area electron diffraction (SAED) pattern of the bimetallic FeCo nanoparticles is represented. The SAED patterns did not show many dots and circles, indicating that the bimetallic FeCo nanoparticles are very fine particles.

To quantitatively measure the amount of FeCo in each sample for measurement of  $M_s$ , elemental analysis was performed on these bimetallic nanoparticles prepared after washing with ethanol and subsequently annealed at 400 ° C for 4 h, in Argon (Ar) atmosphere.

Vibrating sample magnetometer (VSM) is a very common and versatile method of measuring magnetic properties that determines the magnetic

moment by vibrating the sample perpendicular to a uniform magnetic field in between a set of pickup coils. In a VSM, a sample is properly placed inside the detection coils and subjected to mechanical movement. VSM having the sensitivity range of  $1 \times 10^{-7}$  emu to  $1 \times 10^3$  emu and magnetic field up to 7 kOe was used to evaluate the magnetic saturation and hysteresis loops of bimetallic nanoparticles at room temperature with field sweeping from -7 to +7 kOe. This phenomenon is known as magnetic hysteresis. Fig. 3 shows a plot of magnetization versus magnetic field at room temperature for the oxide-free FeCo bimetallic nanoparticles with saturation magnetization of 221 emu/g, where the magnetization hysteresis loop have been appeared in extremely thin S-shape due to the absence of remanence and negligible coercivity. The FeCo nanoparticles, annealing was necessary to achieve higher values of saturation magnetization, indicating that the inter-diffusion of Co into the Fe lattice is a higher energy process. Interestingly, after the annealing step, there is no noticeable change in the size of these nanoparticles.

## CONCLUSION

In conclusion, FeCo bimetallic NPs were synthesized by a facile method using 2-thioctic acid as a protecting agent and  $\text{NaBH}_4$  as a reducing agent. The XRD confirms the FeCo nanoparticles have pure a phase with 7 nm crystalline size. The as-prepared bimetallic nanoparticles have narrow size distribution, excellent crystallinity,

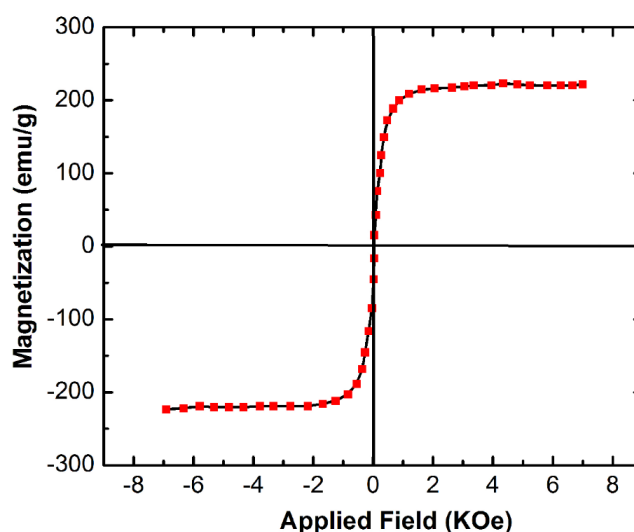


Fig. 3: Room temperature magnetic hysteresis loops of FeCo bimetallic nanoparticles.

and spherical shape. The particle size of these nanoparticles is in the range of 20-30 nm with an optimized room-temperature saturation magnetization of 221 emu/g under controlled-size condition was successfully performed. These small, highly magnetic nanoparticles could have potential applications in engineering applications such as nanocomposite permanent magnets.

#### CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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