

Effect of Temperature on Synthesis and Morphology of Nickel Doped Cobalt Ferrite in Presence of Htab by Co-precipitation Route

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Abstract: Nanoparticles of nickel substituted cobalt ferrite have been synthesized in presence hexa-decyl trimethyl ammonium bromide (HTAB) as a surfactant by co-precipitation route. The particle size was estimated by the full width half maximum (FWHM) of the strongest X-ray diffraction (XRD) peak. The average particle size varied in the range of 22-38 nm; the particle size was controlled via controlling calcination temperature which was in the range of 600 to 900°C. Their morphology structure have been determined by scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis confirms the presence of Co, Ni, Fe and oxygen as well as the desired phases in the prepared nanoparticles. The results of SEM show that the surfactant played an important role in morphology of nanoparticles and a well crystalline single cubic structure of nickel doped CoFe_2O_4 phase was formed through precipitation precursors at pH value of 11.

Key words: Cobalt Ferrite • HTAB • Co-precipitation Method • Calcination Temperature • XRD • SEM

INTRODUCTION

In recent years, ferrite nanoparticles as useful magnetic and electrical properties and their applications in several important technological fields such as magnetic drug delivery [1], magnetic high-density information storage [2] and ferrofluids [3] have been investigated. Cobalt ferrite (CoFe_2O_4) is a well known hard magnetic material having high coercivity and moderate magnetization. These properties, along with their great chemical and physical stability, make CoFe_2O_4 nanoparticles are suitable for magnetic recording applications such as audio and videotape and high-density digital recording disks [4, 5]. While, nickel ferrite is a soft material possesses low coercivity and saturation magnetization. These (soft magnetic) properties make them very promising candidates for various applications in biomedical [6-8]. Conventional techniques for preparation of nanoparticles are included sol-gel processing, evaporation condensation, hot spraying, matrix isolation, sonochemical reaction, laser-induced vapor phase reactions and aerosols. It is considered that the reaction condition as important parameter in determination of magnetic properties of the prepared nanoparticles. Generally, in most types of nanoparticles prepared by these methods, control of particle size and

size distribution is not possible [8]. In order to overcome such difficulties, nanometer size reactors for the formation of homogeneous nanoparticles of nickel doped cobalt ferrite are used. To protect the oxidation of these nanoparticles from the atmospheric oxygen and also to prevent their agglomeration, the particles are usually coated and dispersed in some medium like sodium dodecyl sulfate (NaDDS) or oleic acid [9, 10].

In this research, the process of synthesis of nanoparticle the particle size distributions and particle size are controlled by the use of surfactant in a wide range of temperature. The main advantage of the use of surfactant over other methods is to have well control on the size of synthesized nanoparticle. Besides that, the production of ferrite nanoparticles is relatively easy and the process is straight forward. There is no additional process involved; such as microwave heat and mechanical treatments. The synthesis of nickel doped cobalt ferrite nanopowders using co-precipitation route was considered in the present research.

Experimental: All the chemicals and reagents used in the experiments were supplied by Merck (Darmstadt, Germany) analytical reagent grade. The chemicals were used without further purification. Deionized water was used for the preparation of all the samples. Iron chloride

($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), cobalt chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), hexadecyl trimethyl ammonium bromide (HTAB) ($\text{C}_{16}\text{H}_{33}\text{N}(\text{CH}_3)_3\text{Br}$) and ammonium hydroxide ($\text{NH}_3 \cdot \text{H}_2\text{O}$) were used for the synthesis of nickel doped cobalt ferrites.

Salt solution of 0.2 M iron chloride, 0.1 M cobalt chloride and 0.1 M nickel chloride were prepared in deionized water and then mixed together. A 2.0 M solution ammonium hydroxide was added as precipitating agent. HTAB (0.02 M) was used as surfactant. The reaction was continued at 70°C duration of 6 hours with vigorous mixing at pH value of 11. The precipitated product was then centrifuged at 2500 rpm and filtered to isolate it from the liquid supernatant and then oven dried at 150°C for 8 hours. The brown solid phase was calcined at high temperature ($600\text{--}900^\circ\text{C}$) for duration of 4 hours to get the desired nanoparticles. Prior to characterizations of the prepared nanoparticles, for the removal of any existing impurities, the final products were washed with deionized water and ethanol for several times.

The size and morphology of nanoparticles have been investigated by scanning electron microscope (SEM, Oxford CAMSCAN-MV2300). The crystal structure was characterized by X-ray diffraction (Model: XPERT-MPD, Philips) with Cu K_α radiation over the 2θ range of $10\text{--}70^\circ$ with a step width of $8^\circ/\text{min}$. The accelerating voltage and emission current were 40 kV and 30 mA, respectively. All measurements were performed at room temperature.

RESULTS AND DISCUSSION

The X-ray diffraction patterns of the nickel doped cobalt ferrite with HTAB as surfactant annealed in a temperature range of $600\text{--}900^\circ\text{C}$ for 4 hours are shown the crystallinity of the final product that is illustrated in Fig. 1(a–d). All the peaks in the pattern correspond to the cubic phases. The approximate crystallite sizes of the nanoparticles with HTAB can be calculated by the Scherrer's equation as follows [11]:

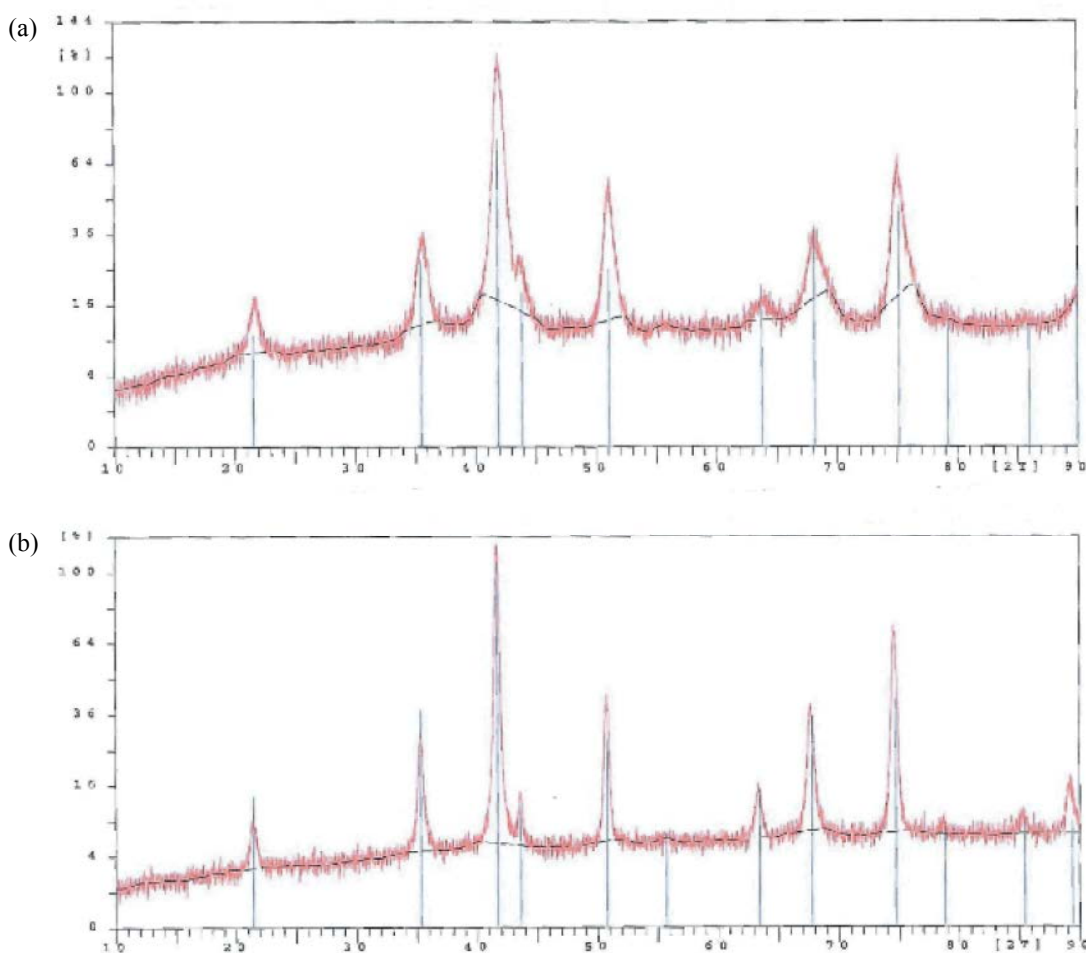


Fig. 1: Continued

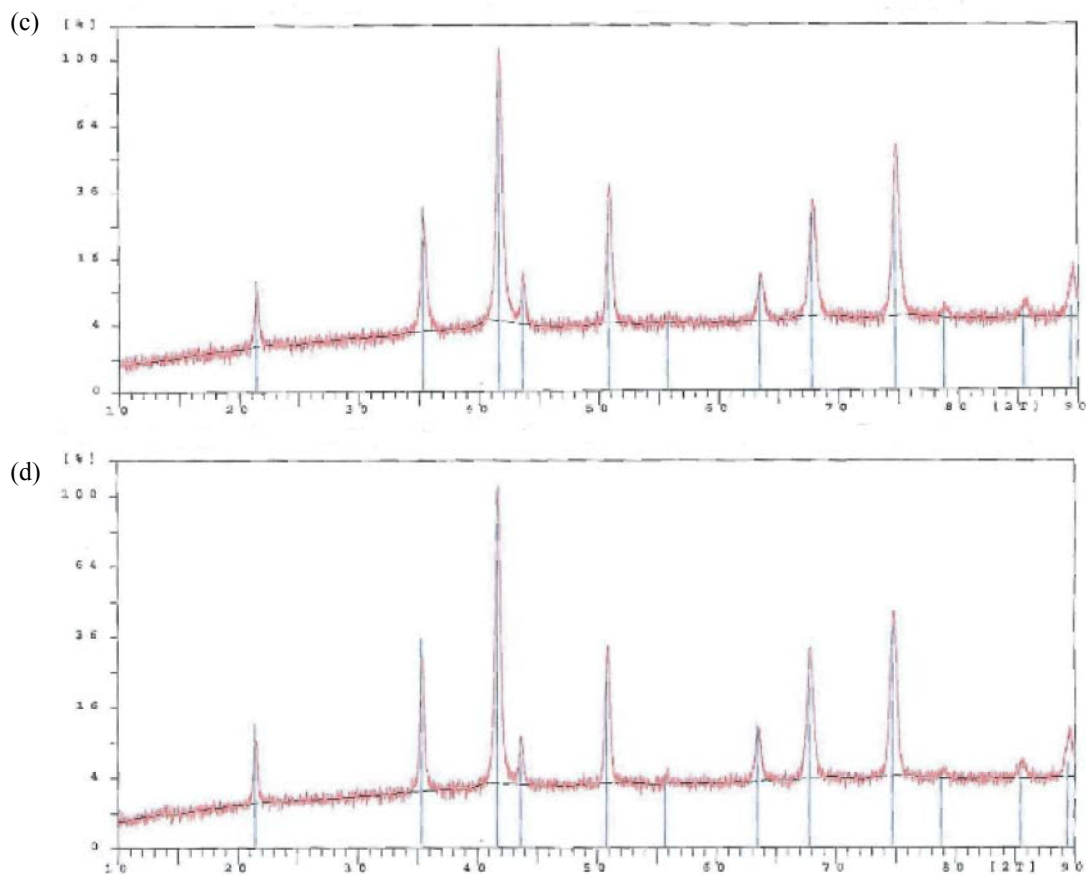


Fig. 1: XRD patterns of the nickel doped cobalt ferrite nanocrystals with TAB at a) 600, b) 700, c) 800 and d) 900°C.

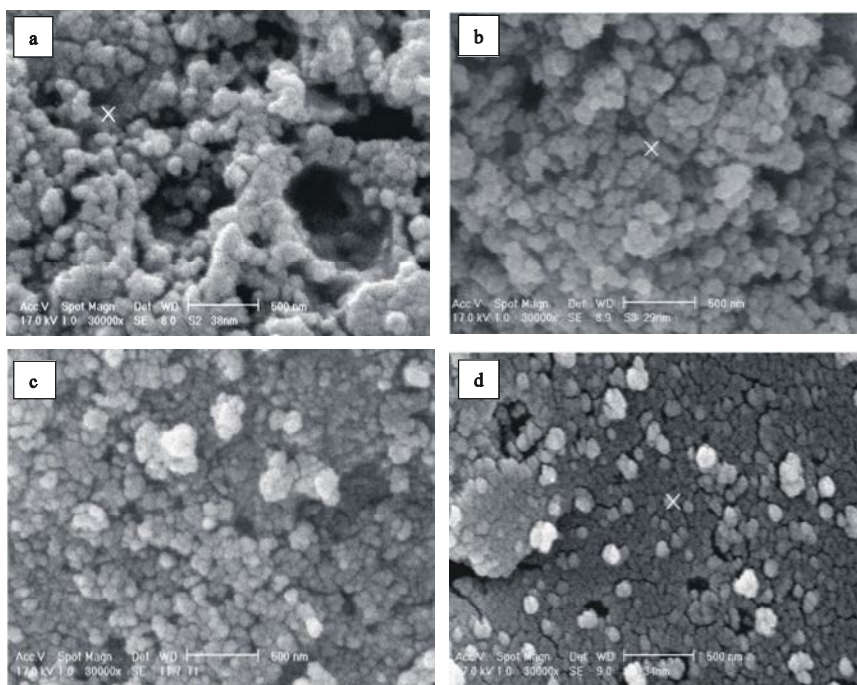


Fig. 2: SEM images of the nickel doped cobalt ferrite nanocrystals with HTAB at a) 600, b) 700, c) 800 and d) 900°C.

$$D_c \frac{k\lambda}{\beta \cos \theta}$$

Where k is so-called shape factor, which usually takes a value of about 0.9 β the breadth of the observed diffraction line at its half-intensity maximum and λ is the wavelength of the X-ray source used in XRD spectrum.

Figure 2(a–d) show the SEM images of the nickel doped cobalt ferrite nanopowders prepared at different temperatures by co-precipitation method. These nanoparticle indicate that sphere-like nickel doped cobalt ferrite nanostructures obtained by this co-precipitation method with HTAB are uniform in both morphology and particle size at various temperature. There is no agglomeration in the prepared nanopowders. In presence of the coated surfactant at pH value of 11, the average sizes of nanoparticles were calculated by Debye-Scherrer's equation to be 22, 29, 30 and 38 nm at 600, 700, 800 and 900°C, respectively.

CONCLUSION

In summary, a simple synthesis route has been developed to fabricate nickel doped cobalt ferrite nanocrystallite with average size between 22-38 nm. The HTAB-assisted co-precipitation route at defined pH value of 11 in a wide range of temperature. The morphology of prepared nanoparticles and structural characteristic were evaluated by SEM images and XRD spectrum, respectively. The SEM images showed that the morphology of grains were regular spherical nanoparticles. Also, there is no agglomeration in final nanopowders. The SEM images also showed that in presence of HTAB at stated experimental conditions homogeneous powders were obtained. The well nickel doped cobalt ferrite has been calcined in a temperature range of 600-900°C for 4hour. The XRD spectrum showed that nanocrystallite sizes decreased when temperature increased.

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