

SYNTHESIS OF NICKEL DOPED COBALT FERRITE IN PRESENCE OF SDS WITH DIFFERENT HEAT TREATMENT BY CO-PRECIPITATION METHOD

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Structural properties of nickel doped cobalt ferrite were synthesized by co-precipitation method with sodium dodecyl sulfate (SDS) as a surfactant at different temperatures. The particle size was estimated by the full width half maximum (FWHM) of the strongest X-ray diffraction (XRD) peak. The average particle size was in the range of 21-36 nm. The particles size was controlled via controlling calcination temperature which was in the range of 600 to 900°C. The morphology of nickel doped cobalt ferrite was investigated. The results showed that a well crystalline single cubic structure of nickel doped CoFe_2O_4 phase was formed through precipitation precursors at pH value of 11. The pH was adjusted by the use of ammonium hydroxide solution.

(Received May 17, 2013; Accepted July 20, 2013)

Keywords: Co-precipitation, Surfactant, SDS, SEM, XRD, Nanoparticle and Cobalt Ferrite

1. Introduction

In the last two decades, inorganic materials with layered structure have been used to improve

the properties of polymers materials with the polymer molecular chain intercalating galleries of adjacent inorganic layers to form nanocomposites, which consist of novel metals, have important applications in medical identification, catalysis, sensors, optics, and electronics, because of the features of their shapes and sizes [1-11].

Research of magnetic nanoparticles has increased in recent years due to their potential in a variety of applications [12] such as genetic screening, biochemical, and toxicity cleansing [13-16], targeted drug delivery vectors for gene therapy, in vivo imaging and contrast agents [14], magnetic cell sorting schemes and spintronic devices [17]. Cobalt ferrite is a well-known hard magnetic material with relatively high coercivity and saturation magnetization while nickel ferrite is a soft material with low coercivity and saturation magnetization. Many of these (soft and hard magnetic) properties make them very promising candidates for different applications in electronic (recording technology) and biomedical [18-20]. A great variety of techniques has been used to obtain magnetic nanoparticles such as milling, co-precipitation, synthesis in reverse micelles and hydrolysis of precursors [21]. It is considered that the reaction condition as important parameter in determination of the magnetic properties of the prepared nanoparticles. Generally, in

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most types of nanoparticles prepared by these methods, control of size and size distribution is not possible [22]. In order to overcome these difficulties and to protect the oxidation of these nanoparticles from the atmospheric oxygen and also to stop their agglomeration, the particles are usually coated and dispersed in some surfactant [23].

In this research, in the process of synthesis the nanoparticles size control and size distribution is obtained by the use of SDS as a surfactant at wide range of temperature. The main advantage of the use of surfactant over the other methods is to have well control on the size of prepared nanoparticle. 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.

2. Experimental

All the reagents used in the experiments were supplied by Merck (Darmstadt, Germany) with analytical reagent grade, and were used without further purification. Iron chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), nickel chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), cobalt chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), sodium dodecyl sulfate (SDS) ($\text{C}_{12}\text{H}_{25}\text{SO}_4\text{Na}$) and ammonium hydroxide (NH_4OH) 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. Ammonium hydroxide of 2.0 M solution was added as precipitating agent. SDS (0.02 M) was used as the surfactant. The reaction was continued at 70°C for 6h with vigorous mixing at $\text{pH}=11$. The precipitated product was then centrifuged at 4000 rpm and filtered to isolate it from the liquid supernatant and then oven dried for 6h at 200°C . The dark brown solid phase was calcined at different temperature ($600\text{-}900^\circ\text{C}$) for duration of 4 hours to get the desired nanoparticles. Before the characterizations of the prepared nanoparticles, the final products were washed with deionized water and ethanol for several times; for the removal of any existing impurities.

The crystal structure was characterized by X-ray diffraction (Model: XPERT-MPD, Philips) with $\text{Cu } K_\alpha$ radiation over the 2θ rang 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. The morphology of nanoparticles was performed by scanning electron microscope (SEM, Oxford CAMSCAN-MV2300). All measurements were performed at room temperature.

3. Result and Discussion

The XRD patterns for the samples of nickel doped cobalt ferrite with SDS as surfactant at different temperature ($600\text{-}900^\circ\text{C}$) are presented in Figure 1.

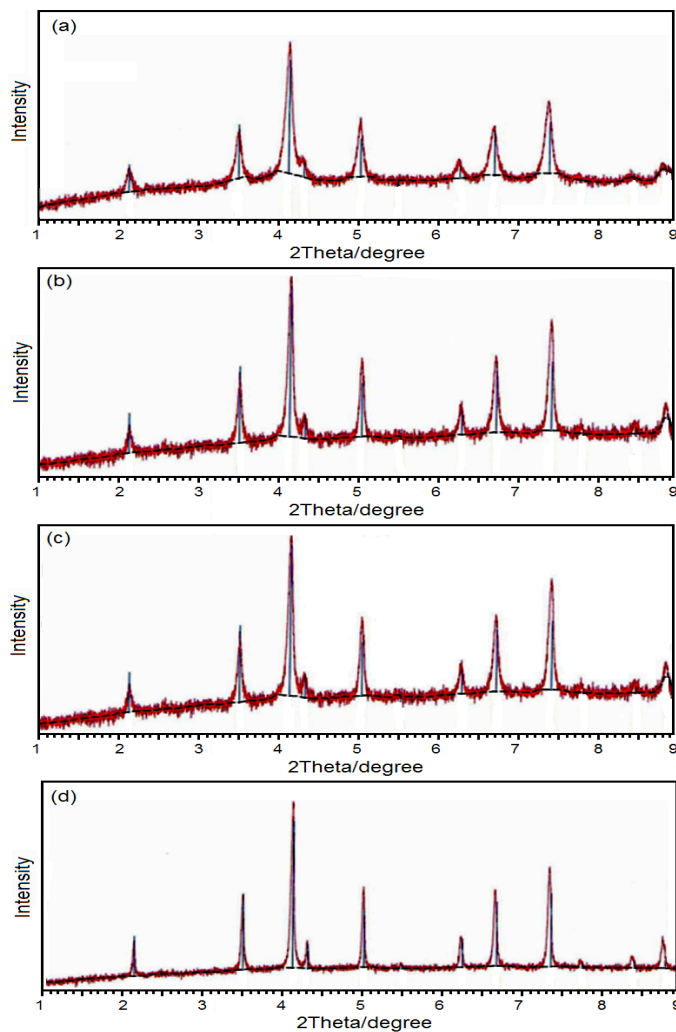


Fig. 1. XRD patterns of the nickel doped cobalt ferrite nanocrystals with SDS at (a) 600, (b) 700, (c) 800 and (d) 900°C.

From the XRD data, the crystallite size (D_c) of the final nanopowders was calculated to be between 21 and 36 nm, using the Debye-Scherrer's equation [24].

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

Where k is the so-called shape factor, which usually takes a value of about 0.9, β is the full-width at half maximum (FWHM) of the peak and λ is the wavelength of the X-ray source used in the XRD. Also, θ is the Bragg's angle. The strong and sharp reflection peaks suggest that the as-product nanocrystals are well crystallized.

The morphology of the nickel doped cobalt ferrite nanopowders were determined by SEM. The SEM images of final products are shown in Fig. 2.

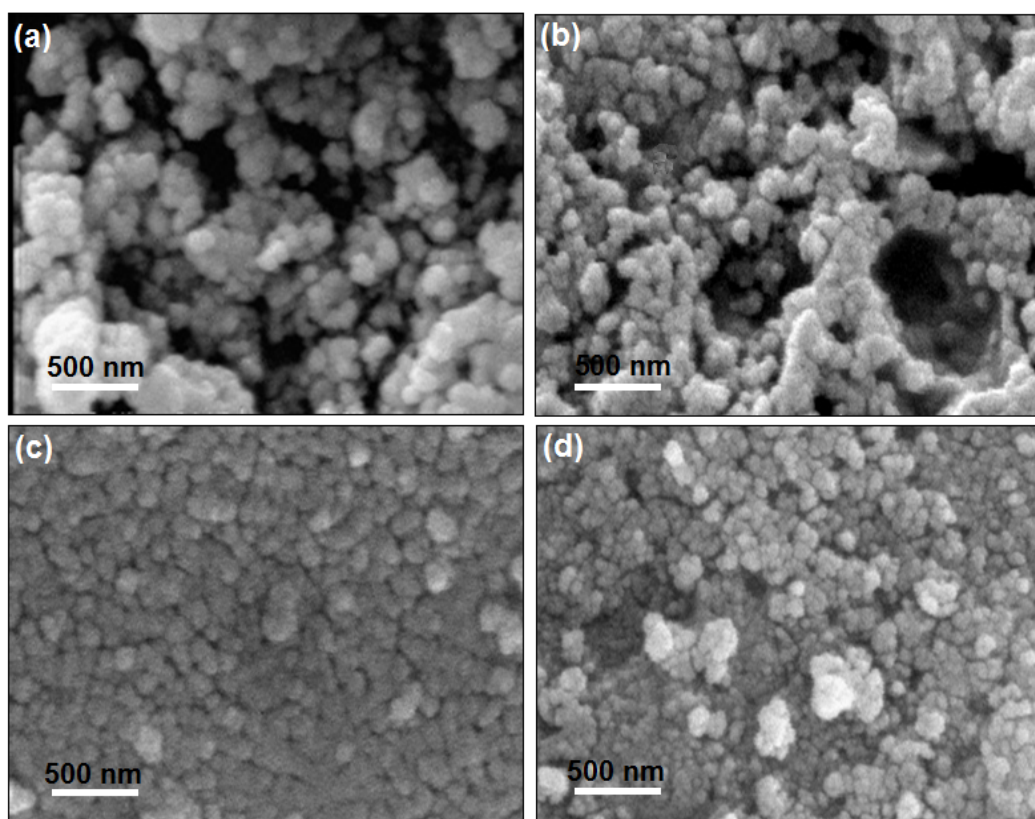


Fig. 2. SEM images of the nickel doped cobalt ferrite nanocrystals with SDS at (a) 600, (b) 700, (c) 800 and (d) 900°C.

These indicate that sphere-like nickel doped cobalt ferrite nanostructures obtained by this co-precipitation method with SDS are uniform in both morphology and range of particles size at different temperature. There is no agglomeration in prepared nanopowders. The average sizes were calculated by Debye–Scherrer's equation to be 21, 28, 31 and 36 nm at 600, 700, 800 and 900°C in present SDS at pH=11, respectively.

4. Conclusion

A simple synthesis route, the SDS-assisted co-precipitation method at defined pH value of 11 in a wide range of temperature, has been used to fabricate nickel doped cobalt ferrite nanocrystallite with average size between 21–36nm. Structural characteristic and morphology were studied by XRD and SEM, respectively. The well nickel doped cobalt ferrite has been calcined in a temperature range of 600-900°C for 4 h. XRD patterns showed that nanocrystallite sizes are decreased when temperature increased. The SEM images showed that grains were regular sphere shaped nanoparticles. Also, there is no agglomeration in final nanopowders. The SEM images showed that homogeneous powders were obtained in presence of SDS at stated experimental conditions.

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