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Study on Synthesis and Characterization of $CoFe_2O_4$ Nanoparticles

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Abstract. This work reports the use of micro emulsion method to prepare $CoFe_2O_4$ nano-particles with sodium hydroxide as an alkaline solution and ferric salt. More vigilance is required while obtaining consistent products as the ferrous salt is unstable in nature. Nano particles developed were characterized for size and morphology by X-ray diffraction (XRD) & Scanning electron microscope (SEM). SEM examined the particle size distribution of 40-70nm. It is difficult to obtain uniform-sized nano-particles as their preparations are mostly done in bulk aqueous media in which it is hard to control nucleation and grain growth.

Keywords: Nanoparticles, Synthesis, Characterization

1. Introduction

Nanotechnology deals with design, synthesis and application of nanostructures and explores the relationships between physical properties, phenomena and structural dimensions [5]. Our aim in this paper was to develop (cobalt ferrite) $CoFe_2O_4$ nano particles which have variety of applications from nanoscale electronics like gauges, sensors and optics to nano-biological systems and nano-medicine[1].Till date, most popular method for the preparation of $CoFe_2O_4$ nano particles are coprecipitation method and Micro emulsion method[2-4]. The method of preparation and substrate utilization affect the final shape of the particle [6].Size range of particles obtained from coprecipitation method [8, 9, 12] is 11-45nm.The crystalline size of 10-12 nm was prepared via polyol method [10].Average particle size of Cobalt ferrite nano-particles by thermolysis method was 45nm [11].Various synthesis methods can be grouped into two categories: thermodynamic equilibrium approach and kinetic approach. In the thermodynamic approach, synthesis process consists of (i) generation of super saturation (ii) nucleation and (iii) subsequent growth. In the kinetic approach, formation of nano-particles is achieved by either limiting the amount of precursors available for the growth or confining the process in a limited space such as aerosol synthesis or micelle synthesis. Micro emulsion method for synthesis of $CoFe_2O_4$ nano particles is observed to be complicated, hence more care is needed while obtaining consistent products [5]. Micro emulsion Method using ferric and cobalt salts is promising technique for synthesis of cobalt ferrite nano-particles[14].

2. Materials and Methods

2.1. Materials

All chemicals are reagent grade and used without purification. Cobalt chloride hexa hydrate ($CoCl_2 \cdot 6H_2O$)(98 %,merk),Ferric chloride hexa hydrate ($FeCl_3 \cdot 6H_2O$) (96 %, qualigens), Oleic Acid (88%,merk),Sodium hydroxide (NaOH)(97%,qualegens),Ethanol (99.9%,Sdfine).

2.2. Method

Iron chloride solution, 0.4M, was prepared by adding 10.58gm of iron chloride in 100ml of distilled water. 4.72 gram of cobalt chloride was added in 100 ml of distilled water to get 0.2M cobalt chloride

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solution. 3.25ml iron chloride solution and 25ml of cobalt chloride solution were mixed with distilled water. Addition of distilled water avoids the formation of crystals. 2.3M sodium hydroxide was added drop wise with continuous stirring and pH was maintained at 12. 40ml of oleic acid as a surfactant was added in the liquid salt solution[14]. After adding oleic acid, liquid salt solution gets precipitated. At reaction temperature of 85^{o}C, precipitated liquid was stirred continuously for 2hrs.The liquid is slowly allowed to cool at room temperature. Liquid and solid precipitate separated with the help of filtration. Precipitate was washed with distilled water and ethanol (1:1) to remove sodium and chlorine compounds. Supernatant liquid were decanted and rest liquid in the particle was removed by centrifuge for 20min at 3500rpm. Particles synthesized were black in colour. Product particles were dried at 80^{o}C for 24hrs.Particles were ground into fine powder and annealing was carried out at 900^{o}C for 14hrs.Synthesized particles were obtained and were studied by XRD and SEM.

3. Results

Black colored $CoFe_2O_4$ nanoparticles were synthesized from the procedure as described above. The X-ray diffraction pattern (fig.1) of synthesized particles examined that the final product is CoFe2O4 with the expected inverse spinel structure.

Nanoparticles were analysed for size using XRD.Following specifications were used:

K-Alpha1 [Å] 1.54060

Generator Settings 40 mA, 45 kV

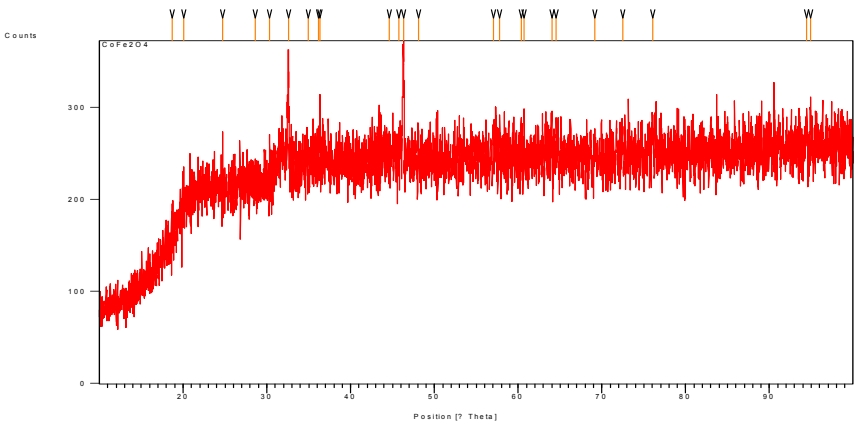
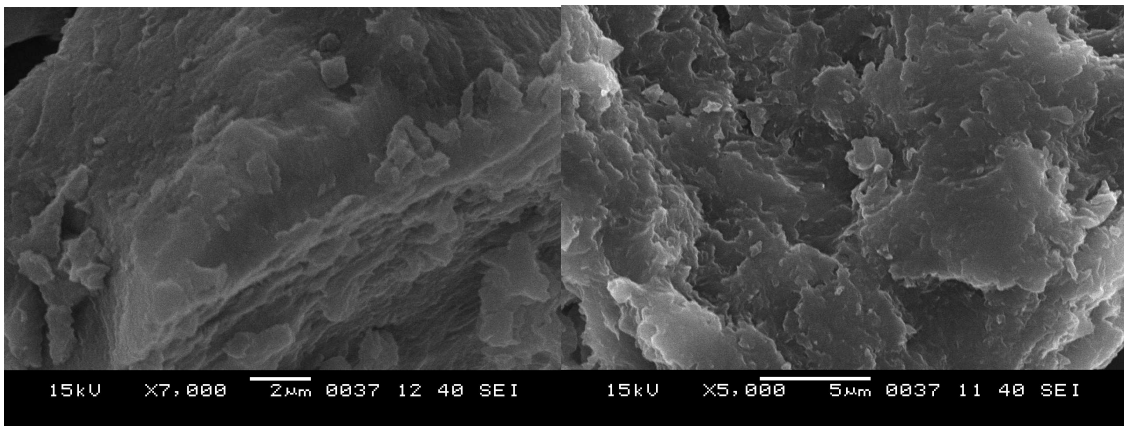


Fig. 1: The graph obtained from the XRD

Intensity peak was obtained at 32.5797^{o} at X-axis.The corresponding value of particle for highest intensity was obtained 42.32 nanometers. Morphology and shape of nanoparticles have been studied using scanning electron microscope. It is clearly indicating that agglomeration of nanoparticles is due to nature and forces between them.



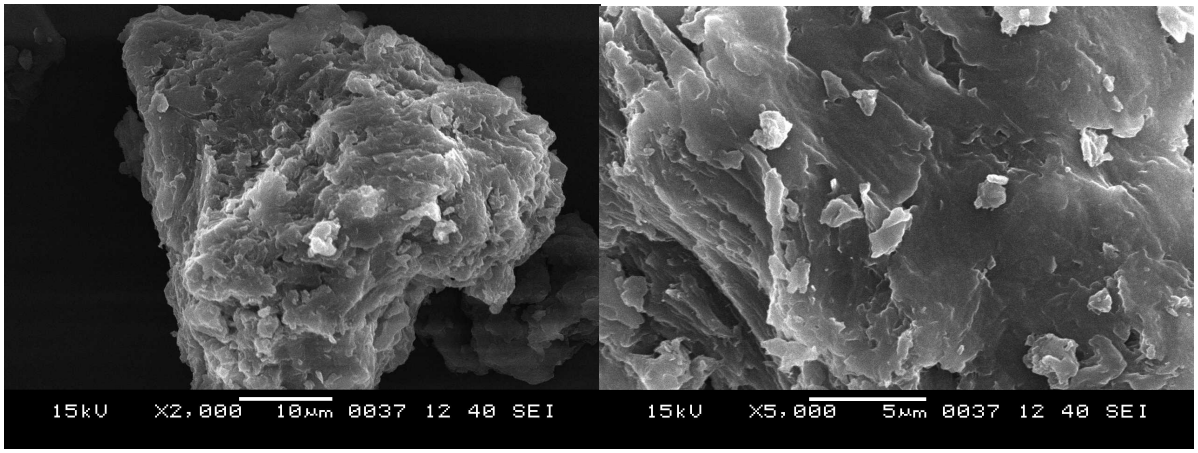


Fig. 2: Scanning Electron microscope image of cobalt ferrite nanoparticles

4. Conclusions

In this project, we have presented the synthesis of $CoFe_2O_4$ nanoparticles in range of 40-70 nm. The size of nanoparticles was measured by XRD, shape and morphology using SEM and was in very good agreement with each other. Frequent washing with ethanol and water can avoid agglomeration occurred initially. It is evident that desired particle size and its distribution may be achieved by controlling the rate of reaction, the annealing temperature and time period.

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