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Synthesis and Characterization of Monodisperse Fe-Co-Ni Colloidal Nanoparticles by Polyol Method

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Abstract

The monodisperse Fe-Co-Ni trimetalic powder has been synthesized by polyol method. The synthesized nanoparticles were characterized by scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS) and Fourier transform infrared spectroscopy (FT-IR).The morphological features as studied using SEM revealed that the nanoparticles were agglomerated, crispy with porous.

Keywords: *Nanoparticles; Polyol method; Characterization; SEM; FTIR.*

1. INTRODUCTION

With the development of nanomaterial's and nanotechnology, more and more efforts have been directed towards large-scale synthesis of nanoparticles in recent years due to their potential applications in many areas. In such uses, nanoparticles with different purity, size, shape and structure will greatly influence the ultimate performance of the devices, accordingly, preparation of nanoparticles with desired quality and low cost by convenient method in large scale is of great importance (Huzhi Wang et al. 2008)

Since the innovation of Nano chemistry in past decades, numbers of materials in Nano-scale have been synthesized via many methods. Nano materials have been widely used in various fields, such as photo electronic, recording materials, catalysis, sensors, ceramic materials, etc., due to their special structures and properties (NowsathRifaya et al. 2012).

Due to their small dimensions and large specific surface areas, nanomaterials have attracted the scientific community in the recent days owing to their potential use as high density magnetic recording systems, catalysis, Nano electronics, nanoscale optics, magnetic fluids, biomedicine and biotechnology. Currently, much attention has been focused on the preparation of magnetic Nanomaterial's because of their potential application in Ferro fluids, advanced magnetic- materials, catalysts, optical and mechanic devices, high density magnetic recording media and medical diagnostics (Alagiri et al. 2011).

In particular, magnetic nanomaterial exhibit very high magneto resistance, large coercivities, high curie temperatures, and low saturation magnetization (Ravi Eluri and Brian Paul, 2012). Consequently, magnetic nanomaterials have attracted much attention in sensors, imaging (Frey et al. 2010), magnetic storage, and ferro-fluids applications (Xia et al. 2010).Several methods have been developed for the preparation of nanoparticles, including gas condensation process (Ho Chang and Ming-HsunTsui 2008), thermolysis(Capezzuto et al. 2009), electrospining method (Chum-Rong Lin et al., 2009), wet chemical method (Sahoo et al., 2009), sono - chemical reduction, metal vapour. In this present work we reported some experimental

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synthesis, chemical reduction (Zhang Qui-li et al. 2010) vacuum vapour deposition, radiation methods, micro emulsion techniques and laser ablation (Jin Zhang et al. 2008), sol gel method (Alagiri et al. 2011) and liquid polyol method (Wei Cai and JiaquiWan, 2007 andHaris et al. 2010).

In this present work we reported some experimental results concerning the synthesis of Fe-Co-Ni nanoparticles obtained by a chemical method called the polyol process. In the polyol process the liquid polyol acts as the solvent of the metallic cations. The solution is heated to a given temperature reaching the boiling point of the polyol for less reducible metals (Ciracet al. 2008).

2. MATERIALS AND METHODS

Cobalt(II) acetate tetrahydrate, nickel(II) acetate tetrahydrate, Iron(II) chloride tetrahydrate, Oleic acid, 1,2-propanediol, dihydroxydiethylether, 1,2-ethane diol, $AgNO_3$, NaOH are of high purity and used without further purification. The Fe-Co-Ni nanoparticles are prepared by reductive thermal decomposition of Co(II) acetate tetrahydrate and nickel(II) acetate tetrahydrate and Iron(II) chloride tetrahydratein the presence of 1,2-propanediol as a reducing agent and a mixture of surfactants such as oleicacid, 1,2-ethanedioland dihydroxydiethylether.

In a typical procedure, 0.13molL ^{1} ofIron(II) chloride tetrahydrate, 0.043 $molL^{-1}$Co(II) acetate tetrahydrate and 0.043$molL^{-1}$ nickel(II) acetate tetrahydrate are added in the 250 mL round bottom flask. 0.25$molL^{-1}$ of NaOH in 1,2-propanediol (1.2 mL) as a reducing agent, 50mL of dihydroxydiethylether in pinch of $AgNO_3$, 1,2-ehthanediol (50 mL), Oleic acid (2 mL) as surfactants are added to it. The solution containing all the reactants and the nucleating agent was slowly heated up to the boiling point under megnetic stirring at 250 rpm and then maintained at this temperature for 2 hours. After cooling, the precipitate of metallic powder was separated, sonicated and washed several times with ethanol, water and acetone and finally dried with under hot air oven in the convection mode at 200^0 C.

The morphology and size distribution of the magnetic nanoparticles were determined by scanning electron microscopy (SEM) technique (model HITACHI Japan). The compositional analysis was conducted by EDS (Energy Dispersive Spectrometry). The FT-IR spectra of the dried powder samples were obtained with a SHIMADZU spectrometer in therange of 4,000– 400 cm^{-1}.

3. RESULTS AND DISCUSSION

The SEM images are shown in Figure 1a, 1b, 1c. The observed larger particles exhibited numerous spherical perturbances on the surface, suggesting that they were formed during the precipitation process through fusion of much smaller particles. The prepared products were agglomerated from few microns to a few tens of nanometers, fluffy and porous (Tahvildari et al. 2012). In Figure 1c shown that some e of the nano rod like structures were performed that the growth conditions. It can be observed that the mean diameter of the nano rod varies between 426 nm to 438 nm.

The EDS analysis of the two single particles are shown in figure 2. This provides the confirmation of presence of Fe, Co and Ni in the single particle. The average composition of each nanoparticle is found to be Fe (=1.58 at. %), Co (=34.57 at. %) and Ni (=28.83 at. %). Apart from Co and Ni peaks, carbon and oxygen peaks are also observed in the EDS spectrum.

The FT-IR spectrum of the sample Figure 3 indicates that the bond has been formed with the oleic acid and the metallic core. The IR bands in the 2854-2924 cm^{-1} region arise from the *vs*($-CH_2$) and*va*($-CH_2$) stretching of the oleic acid carbon chain. A broad *v*(OH) stretch at 3414cm^{-1} is present as well. The two signature bands at 1635 cm^{-1} and 1462 cm^{-1} thatcorrespond to *vs*(C=O) and *va*(C=O) indicating a bond has formed between the particleand the capping agent are present as well. All other bands below 2000 cm-1 are due to the*v*(C-C) stretch, *v*(C-O) stretches, CH_2 deformations and other motions that are toocomplex to assign and

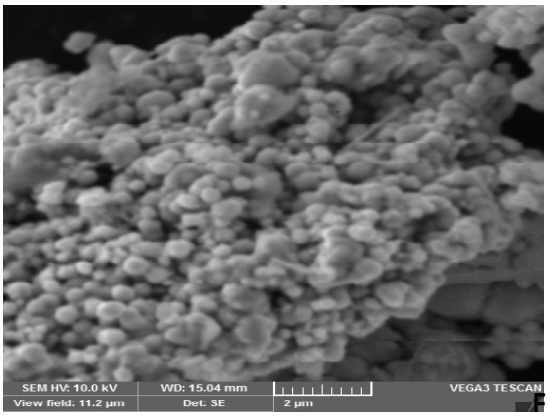

Fig. 1a SEM image of Fe-Co-Ni nanoparticles

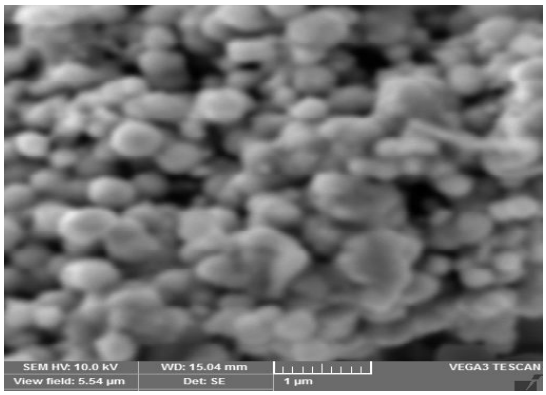

Fig. 1b SEM image of Fe-Co-Ni nanoparticles

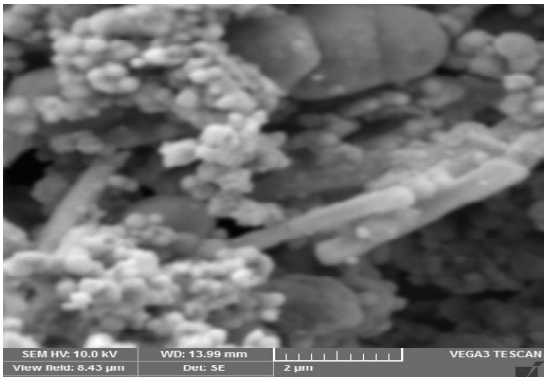

Fig. 1c SEM image of Fe-Co-Ni nanoparticles

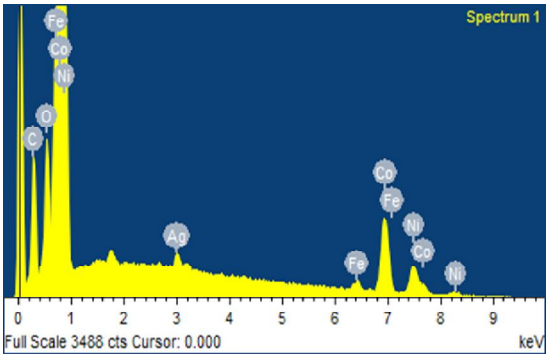

Fig.2 EDS spectrum value of Fe-Co-Ni nanoparticles

indicate the oleic acid chain is still present. The sharp band at1635 cm-^{1} has been attributed to a äH_2O vibration (ä signifies an in phase rock).

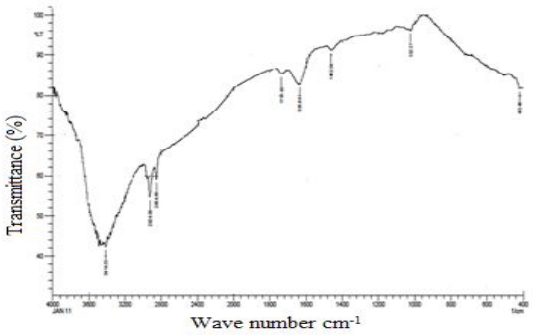

Fig.3 FTIR spectrum of Fe-Co-Ninanoparticles

This result indicates that ethylene glycol remains on this sample, probably adsorbed on the nanoparticles surface, despite the successive ethanol washing steps done after the synthesis of this material. In fact, some bands attributed to ethylene glycol were also detected on the ether containing sample, which should be an indicative that ethylene glycol is also present on the dihydroxydiethylether containing Fe-Co-Ni nanoparticles surface (Giselle et al., 2007).

4. CONCLUSION

The Fe-Co-Ni nanoparticles were successfully synthesized through polyol method. A wide range of Fe-Co-Ni nanoparticle sizes within the micrometer scale was obtained by this method with addition of agglomeration reducing agent. The structure of the synthesized sample was confirmed by SEM studies. FTIR analysis indicates that the carbonyl groups in the oleic acid structure strongly coordinate on the nanoparticle surface. So after synthesizing the sample ethanol washing was needed. Further research is under progress in our laboratory.

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