RESEARCH ARTICLE

MAGNETIC NICKEL NANOPARTICLES: SYNTHESIS AND CHARACTERISATION

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ABSTRACT

Face centered cubic nickel has been synthesized through reduction of nickel chloride by hydrazine at 90^0C.X-ray powder diffraction showed that as-prepared samples were nickel with FCC structure. Scanning electron microscopy indicated that the particles were spherical and EDAX study confirmed the presence of high purity of nickel. Magnetic measurements showed that samples were ferromagnetic. The value of remanent magnetization and coercivity was enhanced while the value of saturation magnetization was decreased when compared to bulk nickel at room temperature.

INTRODUCTION

Synthesis and application of magnetic Nanoparticles with diameters of a few nanometers is a subject of intense research because of their unique properties that make them attractive, both from the scientific value of understanding their properties, and the performance of the existing materials (Fioraniin and Dormann, 1992). In this regard, magnetism of 3d-transition metals like iron, cobalt and nickel is well understood at the bulk level, their nanocounterparts are interesting because at the nanolevel, they exhibit altogether different physical, mechanical, optical, chemical and electrical properties (Lu, Salabas and Schuth, 2007; Holmes, Lyons and Ziegler, 2003). There are several methods, which have been used to synthesize metal nanoclusters: evaporation of metal atoms, microemulsion methods, carbon encapsulation and electrochemical growth (Chen and Wu, 2000; Gong, Li, Zhao and Chen, 1991; Sun and Dong, 2002). A larger number of nanoparticles have been prepared most frequently by the disperse on of performed polymers (Kompella *et al*., 2001) solvent evaporation method (Kwon *et al*., 2001) and ionic gelation method (Calvo *et al*., 1997). All these methods have their own drawbacks. Ni-nanoparticles,in particular being cheap, need mild reaction conditions for high yields of products in short reaction times. We report herein a novel synthesis of Ni-nanoparticles and their characterizations.

Experimental

Ethylene glycol was supplied by Merck, India. Nickel chloride hexahydrate and hydrazine hydrate were obtained from Mumbai. Sodium hydroxide (NaOH) pellets was obtained from Loba Chemie. The deionized water was used throughout the experiment. Nickel sample was prepared by the methods reported earlier (Wu and Chen, 2003), using Nickel chloride hexahydrate as a precursor and hydrazine hydrate as reducing agent. An appropriate amount of $NiCl_2$ is dissolved in10ml Ethylene glycol. Then 10 ml hydrazine hydrate is added to above solution. NaOH are added in sequence and stirred

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well. The complete solution is kept in water bath for 1 h at 90ºC. The resulting black colored solid was washed with ethanol and dried in the room temperature.

RESULTS AND DISCUSSION

Structural Analysis

Fig.1 shows typical XRD patterns of as-prepared nano-powder. Bragg's reflection are observed at 44.56˚, 51.85˚, 76.00˚ representing (111), (200) and (220) planes of FCC crystal structures of bulk nickel. No obvious peaks of Nickel oxides and hydroxides are detected, possibly it is attributed to the observed phenomenon that Nitrogen gas is produced and bubbled up continuously during the reaction. By use of Scherer's equation particle size was calculated and it revealed a crystallite size of about 23.27 nm.XRD analysis of black powders, confirmed formation of nickel in the final product as phase pure nickel nanoparticles (Ni *et al*., 2003; Lanje, Sharma and Pode, 2010).
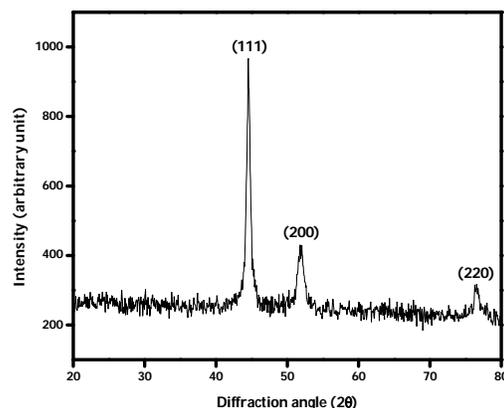
(111)

Fig. 1. XRD of Nickel Nanoparticles

Surface Morphological Analysis

Fig. 2 shows the SEM micrographs of the nickel nanoparticles. SEM images of as prepared nickel nanoparticles revealed formation of spherical particles with homogeneous size distribution around 23.27 nm. The crystallite size calculated from SEM analysis is quite agreement with that of crystallite size calculated from XRD analysis. The reduction process must have been started by hydrazine because hydrazine is a powerful reductant that can reduce nickel quickly from the solution. Due to this fact the similar nickel particles with smooth surface were obtained. EDAX analysis is used to analyze the ingredients of its composition, indicating that the fabricated Nanoparticles contain elements of Ni and O. EDAX analysis (Fig.3) result shows that in sample the weight percentage of O is 1.62% only, whereas that of Ni is as high as 98.38 % .Ethylene glycol might formed as a protective layer on the Ni Nanoparticles and it protects Ni from being oxidized. The oxygen detected could be attributed to partial oxidation of the Nanoparticles which is due to the presence of some residual solvent or during the handling of the sample.
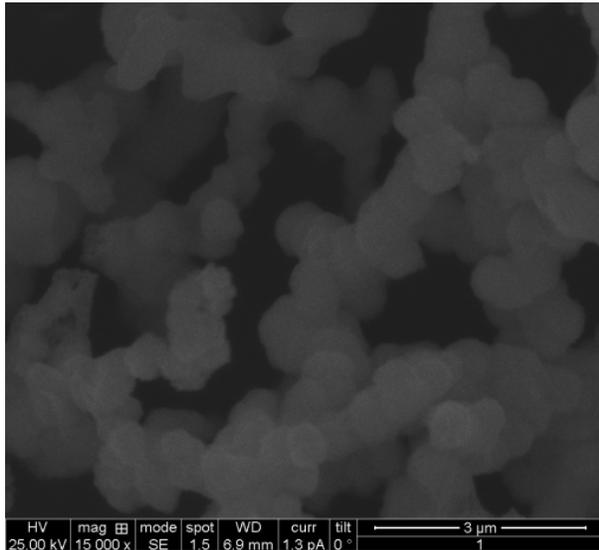

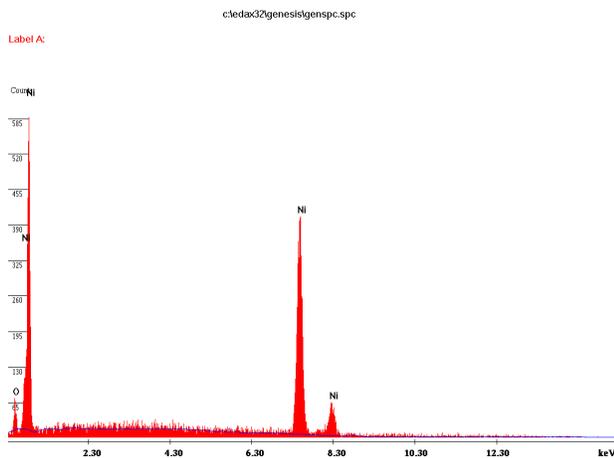**Fig. 2. SEM images of Nickel Nanoparticles**

Fig. 3. Edax Spectra of Nickel Nanoparticles

Optical Spectra Analysis

Fig. 4 is the UV-vis spectrum of the as- synthesized Nickel Nanoparticles. A strong absorption in the UV region is observed at wavelengths about 263 and 265 nm. The energies of the quantum states of Nickel molecules are somewhat spread out due to constant collisions with the surrounding ethylene glycol molecules. Therefore

the Nickel particles absorb photons spread over a range of wavelength and the UV spectrum acquires the shape of a smooth and continuous absorption peak. The property of strong UV absorption should be attributed to the high purity and perfect crystallinity of the as-synthesized Ni Nanoparticles. The quantum size confinement of the crystallites is evident from the blue-shift of the absorption edge in the UV-visible absorption spectrum (Hwang *et al.*, 1997). Fig. 5. shows optical transmittance spectra of the nickel Nanoparticles. The nickel Nanoparticles are transparent in a large range of wavelength.
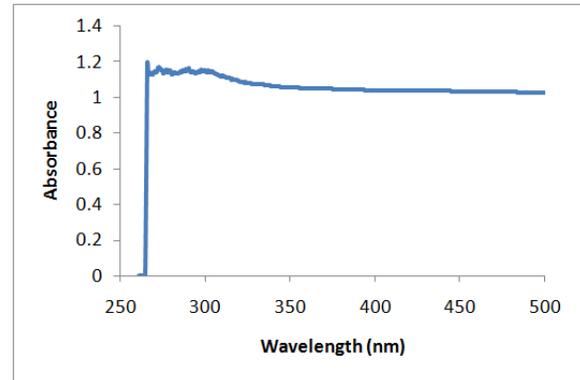

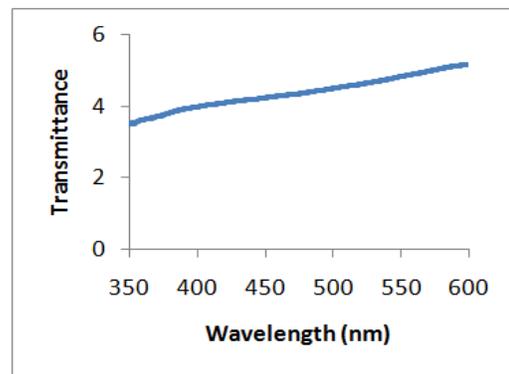**Fig. 4. Optical absorption spectra of Nickel Nanoparticles**

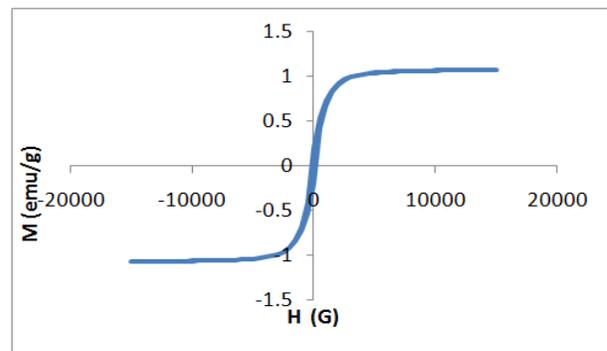**Fig. 5. Optical Transmittance spectra of Nickel Nanoparticles**

Fig. 6. M-H loop measured at RT for obtained Nickel Nanoparticles

Magnetic Measurement Analysis

In our work, magnetic properties of the samples were investigated at room temperature using a vibrating sample magnetometer. Fig.6 shows the hysteresis loops of the samples. It can be seen that the saturation magnetization (M_s) is about 42.73 emu/g, remanent magnetization (M_r) is 8.06 emu/g, and coercivity (H_c) is 162.02 G, which indicates that the nanoparticles are ferromagnetic at room temperature. Compared to the Values of bulk nickel (M_s - 55 emu/g, M_r = 2.7 emu/g, H_c = 100 G, 300 K) (Hwang *et al.*, 1997), the value of M_r and H_c was enhanced while the value of M_s was decreased,

which was consistent with the earlier reports (Shafi and Balogh, 1998). The M-H curves clearly shows existence of hysteresis loop with ferromagnetic properties. This behavior can be modeled as a single giant magnetic dipole moment. At low temperatures, this moment is pinned along one of the energetically favorable 'easy' (crystalline) axes of the particle, therefore exhibiting ferromagnetic behavior. This may be attributed to the presence of ethylene glycol layer on the surface of the Nanoparticles. The decrease in M_s might be due to the decrease in particle size and the accompanied increase in surface area. Furthermore, the magnetic molecules on the surface lack complete coordination and the spins are likewise disordered. This phenomenon is more significant for Nanoparticles due to their large surface to volume ratio and may be another factor that leads to the decrease in M_s. As suggested above, a protective layer from ethylene glycol might be formed on the particle surface and this leads to the decrease in M_s (Szu-Han Wu and Dong-Hwang Chen, 2003). By decreasing the particle size of a material to nanometer scale, its magnetic structure will change from a multi-domain to a single domain configuration. Accordingly, the magnetization process will go from domain wall motion to the mechanism of magnetization rotation, which would lead to the increase of its coercivity (Cullity, 1972). Another factor which leads to increase in H_c may be due to their spherical structure having high shape anisotropy and due to the appearance of FCC Ni phase. The reason for these magnetic values changes may be attributed to the decrease in particle size and the increase in surface area.

Conclusion

Ni Nanoparticles were synthesized in ethylene glycol by chemical reduction method using nickel chloride as solvent, hydrazine hydrate as reducer and NaOH as catalyst. XRD analysis indicated the presence of FCC crystal structure of nickel. SEM analysis showed formation of spherical particles. The quantum size confinement of the crystallites is evident from the blue-shift of the absorption edge in the UV-visible absorption spectrum. The spherical Nanoparticles showed ferromagnetic character at room temperature indicating the magnetic properties varied as a function of decrease in particle size.

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