

Liquid-Phase Synthesis of Nickel Nanoparticles stabilized by PVP and study of their structural and magnetic properties

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ABSTRACT

We have synthesized nickel nanoparticles using nickel chloride as a precursor in ethanol using PVP (Poly Vinyl Pyrrolidone) as a surfactant and hydrazine hydrate as reducing agent at 60 °C in a facile manner. The structural analysis showed that particles are face-centered cubic and monodisperse within the PVP matrix with average size about 3 nm. The magnetic analysis shows the superparamagnetism of the single-domain nickel nanoparticles with the blocking temperature (T_b) exists around 14 K with clear hysteretic effect observation below this blocking temperature. Copyright © 2011 VBRI press.

Keywords: Nanoparticles; magnetic materials; electron microscopy; superparamagnetism.



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Introduction

Novel size and shape dependent physical and chemical properties are of great scientific interest especially in single domain range. Regardless of its size, bulk material should have constant physical properties, but as material size decreases to nano scale regime, the surface effects (due to increase surface to volume ratio) become even more important in determining the various properties of the material like quantum confinement effect, surface plasmon resonance and superparamagnetism (SPM) [1-7]. In magnetic nanoparticles as size decreases from bulk to nanoscale, the overall magnetic moment tends to be confined from multi-domain to single-domain. In single-domain range, the magnetic anisotropy energy per particle responsible for holding the magnetic moment along certain directions becomes comparable to the thermal energy, the superparamagnetic phenomenon is observed. The temperature at which this phenomenon occurs is known as blocking temperature and since it is a size dependent property, so knowing its value is of practical importance in various technological applications such as magnetic storage media [8], sensors [9], target drug delivery [10], magnetic resonance imaging [11], magnetic inks [12] etc.

For controlling the size and shape of the nanoparticles various physical and chemical synthetic routes were developed. An important feature in the production of nanoparticles is the ability to keep them physically separated from each other preventing irreversible aggregation especially in magnetic nanoparticles which tends to aggregate due to magnetic dipole-dipole attractions to reduce their surface energy. The aggregation of nanoparticles can be eliminated by steric (coating the particles with a surfactant layer) or ionic (inducing opposite charge on the particle surface) repulsion [13-15]. Polymers such as dextran or polyethylene glycol, low molecular weight compounds such as citric or oleic acid or inorganic materials such as silica or gold have been reported to coat the magnetic nanoparticles in order to prevent irreversible aggregation. The surface coating does not only protect the particles from aggregation, but it can also affect their size and polydispersity, hydraulic diameter, shape and the total effective moment of the nanoparticles [16, 17]. However, to obtain non-agglomerated nanoparticles with a well-controlled mean size and a narrow size distribution is still a challenge.

Metal nanoparticles, especially nickel, have attracted much attention because of their use in numerous practical applications, such as magnetic storage materials [8], magnetic inks [12] and catalysts [18]. Numerous physical and chemical methods have been used to produce high-quality nickel nanoparticles, such as metal evaporation-condensation [19], electrochemical methods [20], metal salt reduction [21], ultrasound irradiation [22], ultrasound spray pyrolysis [23], polyol methods [24] and neutral organometallic precursor decomposition [25, 26]. Generally, chemical reduction methods have the advantage of simplicity, precise control of the size and low cost compared with physical methods. The crystal structure of nickel nanoparticles obtained mostly using the above methods are either fcc (face centered cubic) or hcp

(hexagonal close packing). Different crystal structure with various morphologies like nanoparticles, nanorods etc. can affect the overall magnetic properties of the material. Y. Chen et al. [27] observed that magnetization of the fcc nickel nanoparticles increases rapidly with saturation at around 10 kOe field, whereas that of hcp nickel nanoparticles it increases slowly with no saturation even at 50 kOe. N. Cordente et al. [28] studied the magnetic properties of fcc nickel with various structural morphologies and concluded that the amine-capped structures had magnetization values comparable to the corresponding nickel 'bulk' ones. Thus the knowledge of the exact phase identification along with blocking temperature of nickel nanoparticles is quite important.

In the present study, we report for the first time of the synthesis of monodisperse nickel nanoparticles from nickel salts in a non-aqueous solution using PVP as a surfactant. The nanoparticles size and structure have been characterized by transmission electron microscopy (TEM), thermal gravimetric analysis (TGA), powder x-ray diffraction (XRD), and its magnetic properties were studied using the Superconducting Quantum Interference Device (SQUID) magnetometer.

Experimental

Materials and instruments

The chemicals nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, Mw 237.69, ≥ 98%) and acetone (Mw 58.08, 99%) were purchased from Spectrochem; hydrazine hydrate ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$, Mw 50.06) was supplied by Qualigen; sodium hydroxide (NaOH, Mw approx. 40) was from S.D. Fine-chemicals Limited, Poly Vinyl Pyrrolidone (PVP, Mw 40,000), ethanol, chloroform and methanol was from Loba Chemicals; water was distilled by Elga pure lab equipment (resistivity 18 MΩ cm).

For structural characterization we use Transmission electron microscopy (TEM-Jeol JEM 1010) operated at 80 kV. For preparation of samples for TEM analysis, the nanoparticles were dispersed in chloroform: methanol solution and deposited on carbon-coated Cu-TEM grids (400 mesh). The film on the TEM grid was allowed to stand for a sufficient time to allow the liquid to evaporate. After drying, the specimen was transferred into the microscope column for imaging at different magnifications. Powder x-ray diffraction (XRD) patterns were recorded with a PANalytical X'pert PRO diffractometer with Cu Kα radiation (λ : 1.5406 Å) at room temperature.

TGA was carried out from the dried nickel powder using Water model SDT-Q-600 with the ramping rate at 5 °C/min in the temperature range of 30–800 °C and Super Quantum Interference Device (SQUID) magnetometer (MPMS-XL, Quantum Design) was used to study the magnetic properties of the nickel nanoparticles. For the zero-field-cooled (ZFC) measurement, the sample was cooled to 4 K in the absence of an external magnetic field. A field of 200 Oe was then applied and the magnetization was measured with increasing temperature both. For the field-cooled (FC) measurement, the sample was cooled to 4 K in the presence of a field of 200 Oe, and then the

magnetization was measured with increasing temperature in the same field both for nanoparticles.

Synthesis of nickel nanoparticles

In our earlier work, we have synthesized nickel nanoparticles using cetyltrimethylammonium bromide (CTAB), tetraethylammonium bromide (TEAB) and tetrabutylammonium bromide (TBAB) as surfactants in aqueous solutions [29]. In the present work we are reporting the synthesis of nickel nanoparticles using PVP as a surfactant in non-aqueous medium. A 3.3mM solution of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in 60 mL of pure ethanol was added to a 125 mL round bottom flask at room temperature. Then 0.025 gram of PVP was added to the solution and the mixture was stirred until the total dissolution of PVP. Then 8 mL of hydrazine hydrate followed by 1.7mL of 1M NaOH solution was added under strong magnetic stirring. After this, the solution was heated up to 60 °C with constant shaking and within 5 minutes the solution turned black, indicating the formation of nickel nanoparticles. During synthesis, no additional nitrogen atmosphere was used, as hydrazine itself releases N_2 gas on its decomposition. The solution was then allowed to cool in ambient conditions at room temperature. After cooling, acetone was added in excess in order to precipitate out the nanoparticles. The resulting solution was ultra-centrifuged at the speed of 20,000 rpm and particles were collected and washed several times with a chloroform-methanol (1:1) solution. Finally, the nanoparticles were dried in vacuum and collected for further studies. The flowchart representing the whole process is summarized in Fig. 1.

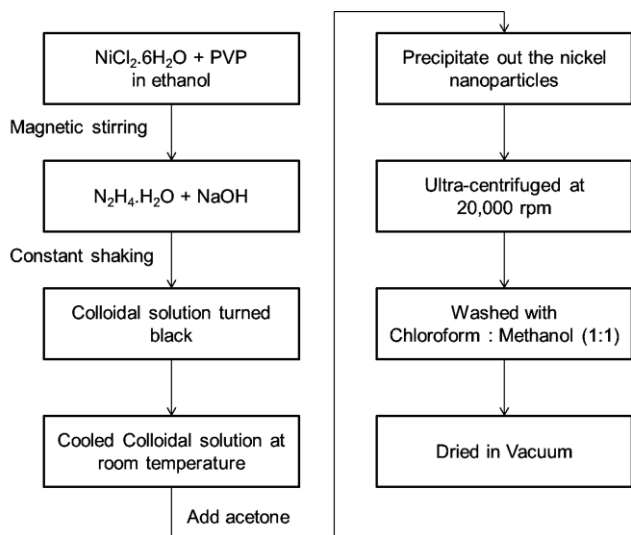


Fig. 1. Schematic representation of the synthesis procedure of nickel nanoparticles.

Results and discussion

Structural characterization

Fig. 2 (a) is a TEM image of nickel nanoparticles showing them to agglomerate in a self-assembled flower like structures possibly either due to magneto dipole attraction, surfactant coating or both. Fig. 2 (b) is the TEM image at higher magnification showing them as nearly spherical in

shape nanoparticles and well distributed within the PVP matrix with the average size of 3 nm.

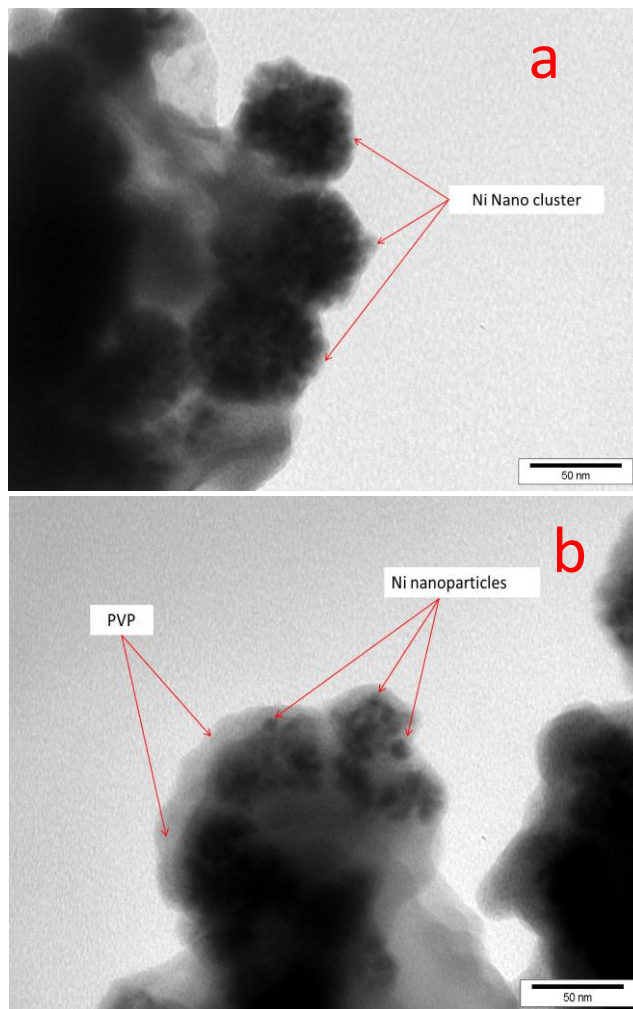


Fig. 2. (a-b) TEM images of as-synthesized nickel nanoparticles.

In the XRD spectra of as synthesized nickel nanoparticles, shown in Fig. 3, no distinct diffraction peaks other than those from fcc nickel can be seen. The broad diffraction peaks are indicative of the ultra-fine grain size of the sample prepared. The XRD pattern of the samples exhibits two characteristics peaks at 44.5° and 51.80° corresponding to the Miller indices (111) and (200) planes of the fcc nickel and does not contain any impurity. Though nickel is easily oxidized, peaks due to its oxide were not observed, which might be due to the fact that the reaction was carried out at appropriate pH, temperature and inactive N_2 was produced by hydrazine concentration which acted as a kind of protective atmosphere. The average grain size (diameter) of the nickel nanoparticles are calculated by the Debye- Scherrer formula as:

$$d = 0.9 \lambda / \sigma \cos \theta \text{ ----- (1)}$$

where λ is the X-ray wavelength, σ is the full-width half-maximum at relative intensity of 100%, and θ is the diffraction angle at that intensity. The size was found to be 2.7 nm which was calculated using above Scherrer's formula by measuring the full width at half maxima of peak

(111). The good agreement between the TEM and XRD data means that each nanoparticle is a single crystal.

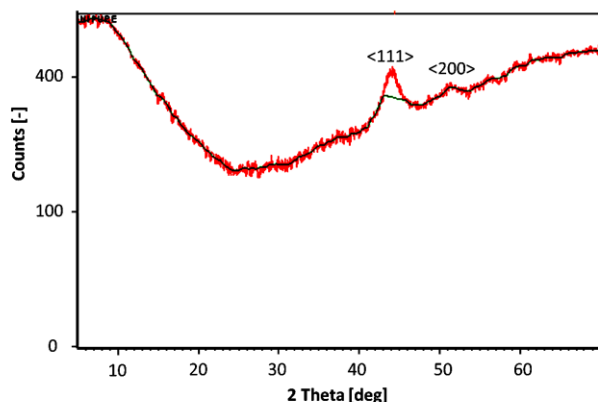


Fig. 3. XRD pattern of the nickel-nanoparticles.

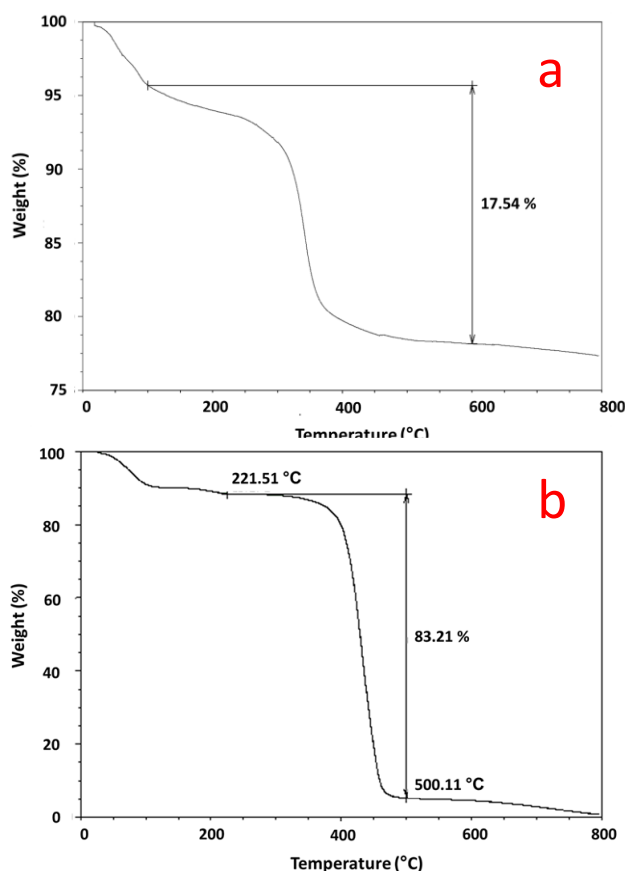


Fig. 4. (a) TGA plot for nickel nanoparticles synthesized using PVP; and (b) for pure PVP at the ramping rate of 5°C/min in the inert atmosphere.

Thermal characterization

The thermal analysis was carried out in order to reveal the presence of PVP on the nanoparticles. The TGA analysis of the nickel nanoparticles and pure PVP in the inert atmosphere is shown in Fig. 4(a-b). The TGA curve in Fig. 4 (a) of PVP coated nickel nanoparticles clearly shows that when the nickel nanoparticles were heated under inert atmosphere they begin to lose weight at 150°C and the total

weight loss is about 17.5% at 600°C which corresponds to the decomposition of the thin PVP coating around nickel nanoparticles. The total weight loss of pure PVP in Fig. 4 (b) is about 83.2% up to 500°C.

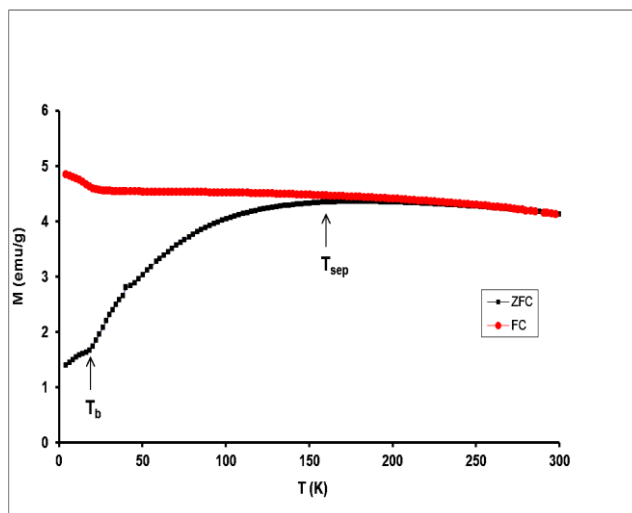


Fig. 5. Temperature dependence of the magnetization (M-T) for the nickel nanoparticles at applied field of 200 Oe.

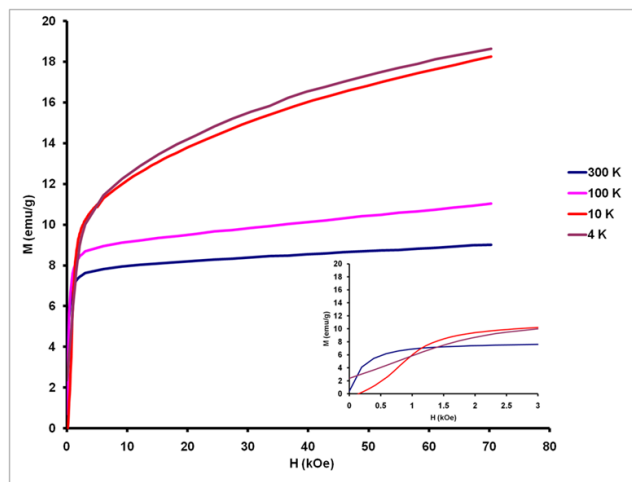


Fig. 6 (a-b) Field dependence of the magnetization (M-H) for the nickel nanoparticles.

Magnetic characterization

The magnetic properties of the nickel nanoparticles were studied by measuring the temperature dependent (M-T) and field dependent (M-H) magnetization curves. The ZFC and FC magnetization curves are plotted in Fig. 5 for the fcc nickel nanoparticles, where the ZFC magnetization monotonically increases with increasing temperature and the FC magnetization almost does not change; the splitting ZFC-FC magnetization curves reach a crossing point around 150 K with blocking temperature (T_b) around 14 K. The blocking temperature is related to the size of the magnetic particles and the magneto crystalline anisotropy constant (K) by the equation:

$$K = 25k_B T_b / V \text{ ----- (2)}$$

where k_B and V are the Boltzmann constant and the volume of a single particle, respectively, and T_b is the blocking temperature. Taking the diameter 3 nm for the nickel nanoparticles and the blocking temperature as 14 K, the magneto crystalline anisotropy constant (K) is $34.15 \times 10^5 \text{ erg cm}^{-3}$. Such values are much larger than the bulk nickel ($K = 5.48 \times 10^4 \text{ erg cm}^{-3}$) [30]. When the particle is too small to accommodate a domain wall (critical radius for single-domain for fcc Ni is about 21 nm [15]), the direction of easy magnetization would change irregularly, causing the phenomenon of superparamagnetism as magnetic anisotropy energy per particle becomes comparable to the thermal energy [15, 30]. Theoretically in superparamagnetism, the hysteretic response is observed below T_b , since the thermal activation is not sufficient to allow an immediate alignment of the particle moments with the applied field. To observe this hysteretic effect, M-H study was done at different temperatures as shown in fig. 6(a), where with decrease in temperature it is difficult to saturate the magnetization of the nanoparticles and fig. 6(b inset) shows the clear hysteretic effect below the blocking temperature. Further in a dead layer theory, the lower saturation magnetization for nanoparticles follows the relation:

$$M_s(\text{nano}) = M_s(\text{bulk}) (1 - 6t/d) \text{ ----- (3)}$$

where t is the thickness of magnetically dead layer and d is the diameter of nanoparticles [25]. Substituting $M_s(\text{bulk}) = 57.6 \text{ emu g}^{-1}$, $M_s(\text{nano}) = 8 \text{ emu g}^{-1}$, and diameter 3 nm respectively, we obtain the dead layer thickness as 0.43 nm. The obtained dead layer thickness in comparison to surface to volume ratio reveals that the obtained nanoparticles can be promising material for magnetic storage media and sensor applications.

Conclusion

In summary, we have synthesized monodisperse nearly spherical nickel nanoparticles in a non-aqueous medium using PVP as a surfactant. Further a detailed size-dependent structural, thermal and magnetic characterization of the nickel nanoparticles has been studied. From the TEM observations, the nickel nanoparticles are of average size of 3 nm which is consistent with the XRD result. The TGA analysis shows the presence of thin PVP coating on the nickel nanoparticles. Observations of the temperature-dependent magnetization (M-T) and hysteresis (M-H) curves of the nickel nanoparticles reveal that the as-synthesized nickel nanoparticles show superparamagnetism (SPM) behavior.

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