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Thermal Decomposition Synthesis of Monodisperse Nickel Nanoparticles: Structural, Morphological and Magnetic Characterization

Ravish Sharma[1], Kash Dev Sharma[2], Rudraksh Tiwari[3], Chandra Prakash[4]
[1,2,3]Assistant professor, Dept. of Physics, B.S.A. (P.G.) College, Mathura, U.P., India
[4]Research Scholar, Dept. of Physics, B.S.A. (P.G.) College, Mathura, U.P., India
Emails: *ravishsharma18jan1983@gmail.in[1], sharma.kashdev@gmail.com[2], drtiwarirudraksh@gmail.com[3], cpdevsharma443@gmail.com[4]*

Abstract
Nickel nanoparticles have attracted considerable attention because of their size-dependent structural, morphological, magnetic, and catalytic properties. In this study, nickel nanostructures were synthesized through the thermal decomposition of a nickel oleate precursor in a mixture of 1-octadecene and oleic acid under a nitrogen atmosphere. The reaction was conducted at 638 K for 80 min with continuous mechanical stirring. The crystalline structure was investigated using X-ray diffraction (XRD), while particle morphology and size were examined using field-emission scanning electron microscopy (FESEM). Magnetic properties were evaluated through field-dependent magnetization measurements at 20 K and 300 K. The XRD pattern exhibited characteristic reflections at 44.78°, 52.16°, 76.72°, 93.23°, and 98.76°, corresponding to the (111), (200), (220), (311), and (222) planes of face-centred cubic nickel, respectively. The average lattice parameter was calculated as 0.350 nm, and the crystallite size estimated using the Scherrer equation was approximately 21 nm. FESEM analysis revealed nearly spherical, weakly agglomerated particles with an average measured diameter of approximately 28 nm, based on the analysis of 100 particles. The saturation magnetization was approximately 35.2 emu g^{-1} at 20 K and 32.5 emu g^{-1} at 300 K. The coercivity decreased from 0.0284 T at 20 K to 0.0039 T at 300 K. These findings demonstrate the relationship between nanoscale structure and magnetic response in thermally synthesized nickel nanoparticles and highlight the importance of controlled synthesis conditions for obtaining nickel nanomaterials with reproducible properties.

Keywords: *Nickel nanoparticles, thermal decomposition, nickel oleate, face-centred cubic structure, X-ray diffraction, FESEM, magnetic properties.*

1. Introduction
Nanomaterials have become an important subject of materials science because their physical and chemical properties can differ significantly from those of their bulk counterparts. At the nanoscale, particle dimensions, morphology, crystallinity, surface chemistry, and interparticle interactions can influence electrical transport, catalytic activity, optical response, and magnetic behaviour. Consequently, controlling nanoparticle size and structure is essential for developing materials with application-specific properties. Nickel is a technologically important transition metal with ferromagnetic behaviour, good electrical conductivity, and catalytic activity. Nickel-based nanomaterials have been investigated for magnetic materials, conductive components, catalytic hydrogen generation, and the catalytic growth of carbon nanotubes [1–3]. Their properties depend not only on their composition and crystal structure but also on particle size, surface oxidation, surface-bound ligands, and the synthesis conditions. Thermal decomposition of metal–organic precursors provides a useful approach for synthesizing crystalline metal nanoparticles. In this method, precursor decomposition, reaction temperature, heating rate, solvent environment, and stabilizing agents influence nucleation and particle growth. Nickel oleate is a suitable precursor for investigating these effects because its thermal decomposition can produce crystalline nickel nanostructures under controlled reaction conditions. Oleic acid can influence particle growth and surface stabilization, while 1-octadecene serves as a high-boiling reaction medium. An inert

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nitrogen atmosphere helps limit exposure to oxygen during synthesis. Nevertheless, the resulting particle characteristics depend on the combined effects of reaction temperature, precursor concentration, reaction duration, stirring, and surface chemistry.[4-5] The structural and magnetic characteristics of nickel nanoparticles have been studied previously, including the dependence of magnetic parameters on particle size. Earlier investigations have also shown that surface effects and protective ligands can influence the magnetization of nanoscale magnetic materials [6–8]. Therefore, correlating crystallographic information with particle morphology and temperature-dependent magnetic measurements is important for understanding the properties of synthesized nickel nanostructures. Recent research has also demonstrated the potential of nickel-containing hybrid materials for energy-storage applications, including plasma-assisted fabrication of PPy-Ni(II) hybrid electrodes with enhanced electrochemical performance [9]. The present study examines nickel nanoparticles obtained through thermal decomposition of nickel oleate at 638 K. X-ray diffraction is used to identify the crystalline phase and estimate the crystallite size, while FESEM provides information about particle morphology and dimensions. Field-dependent magnetization measurements at 20 K and 300 K are used to compare the magnetic response under low-temperature and room-temperature conditions. The study focuses on the relationship between synthesis conditions, structural characteristics, and magnetic behaviour.

2. Experimental Methodology

Nickel oleate was used as the nickel-containing precursor, while 1-octadecene served as the reaction medium and oleic acid was used as a surface-active additive. Nitrogen gas was employed to maintain an inert reaction environment. The quantities and synthesis conditions described below are based on the experimental information provided for this study.For the synthesis of nickel nanoparticles, 2.2 g of nickel oleate was mixed with 21.6 mL of 1-octadecene and 1.3 mL of oleic acid in a five-neck round-bottom flask. The mixture was mechanically stirred for 2 h at room temperature under a continuous flow of nitrogen.After this initial mixing stage, the reaction mixture was heated at a constant rate of 3 K min^{-1} until the temperature reached 638 K. The reaction was maintained at this temperature for 80 min under continuous nitrogen flow and mechanical stirring. Following the reaction, the mixture was allowed to cool to room temperature. Ethanol and hexane were then added to facilitate the separation of the nanoparticles from the reaction medium. The resulting particles were isolated by centrifugation and subsequently dried. The crystalline structure of the synthesized nickel nanoparticles was examined using X-ray diffraction. The diffraction peaks were analysed to identify the crystalline phase and assign the corresponding crystallographic planes. The lattice parameter was estimated from the indexed reflections. The surface morphology of the synthesized nanoparticles was investigated using field-emission scanning electron microscopy. Particle shape, degree of agglomeration, and particle-size distribution were examined from the recorded micrographs. The reported average particle diameter was obtained by measuring 100 particles.The magnetic response of the nickel nanoparticles was evaluated from field-dependent magnetization, measurements at 20 K and 300 K. The resulting hysteresis curves were used to determine saturation magnetization, remanent magnetization, and coercive field. The saturation magnetization was obtained by extrapolating the high-field linear portion of the magnetization curve to zero applied field, as described in the original experimental account. The magnetic parameters at the two temperatures were compared to evaluate changes in the magnetic response.

3. Results and Discussion

3.1.Crystalline structure and phase identification

The X-ray diffraction pattern of the nickel nanoparticles synthesized at 638 K exhibited five prominent reflections at approximately 44.78°, 52.16°, 76.72°, 93.23°, and 98.76°. These peaks were indexed to the (111), (200), (220), (311), and

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(222) planes of face-centred cubic (fcc) nickel, respectively. The observed diffraction pattern is consistent with the formation of crystalline fcc nickel. No additional crystalline impurity peaks were reported in the measured pattern. However, the absence of detectable impurity reflections does not by itself exclude trace impurities, amorphous phases, or a thin surface oxide layer below the detection limit of the measurement. The average lattice parameter obtained from the XRD analysis was approximately 0.350 nm. This value is close to the reported lattice parameter of bulk fcc nickel, although a quantitative comparison requires consistent reference values and consideration of instrumental calibration and peak-position uncertainty. The average crystallite size calculated using the Scherrer equation was approximately 21 nm. This value represents the estimated size of coherently diffracting crystalline domains rather than necessarily the diameter of individual particles. The difference between crystallite size and particle size can arise when particles contain multiple crystalline domains or when diffraction broadening is affected by strain and instrumental contributions.

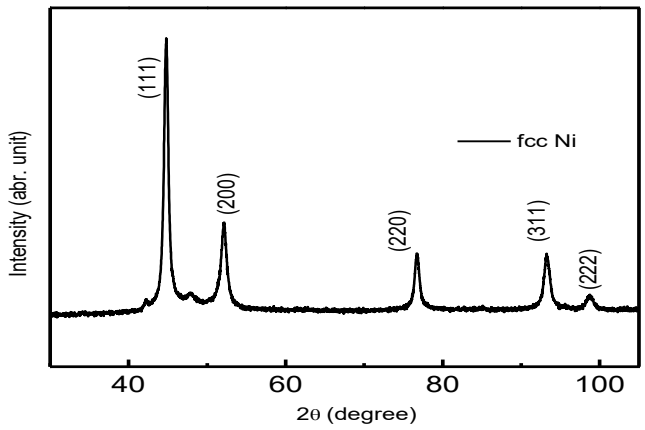


Figure 1 X-ray diffraction pattern of nickel nanoparticles synthesized by thermal decomposition of nickel oleate at 638 K under mechanical stirring

3.2.Surface morphology and particle-size analysis

The FESEM micrographs showed particles with an approximately spherical morphology and a relatively limited degree of agglomeration. T The average particle diameter was reported to be approximately 28 nm based on measurements of 100 particles. The measured particle diameter is larger than the estimated crystallite size of approximately 21 nm. This difference is physically plausible because a particle may contain more than one crystalline domain. However, the available measurements do not independently establish whether the particles are single-crystalline or polycrystalline. The morphology suggests that the combination of nickel oleate, 1-octadecene, oleic acid, and controlled thermal treatment can produce nanoscale nickel particles with approximately spherical shapes. Oleic acid may contribute to surface stabilization by interacting with the particle surface, although direct confirmation of ligand coverage would require complementary surface-characterization measurements. The term monodisperse should be supported by a measured particle-size distribution and an appropriate statistical measure, such as the standard deviation or coefficient of variation. Since only the average diameter and number of measured particles are provided here, the degree of monodispersity cannot be quantified from the available information. As shown in Figure 2 FESEM micrograph of nickel nanoparticles synthesized at 638 K, showing approximately spherical morphology and limited agglomeration.

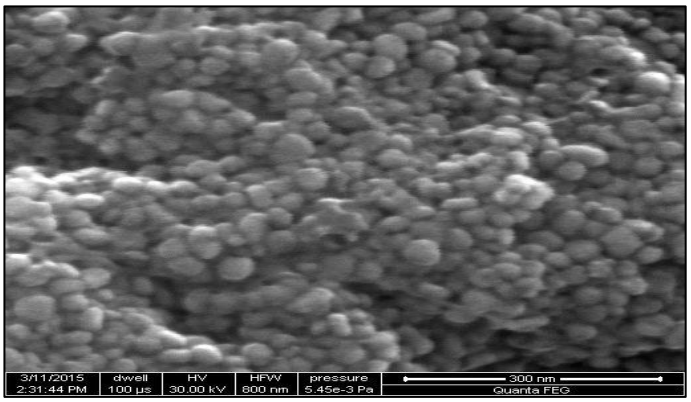


Figure 2 FESEM micrograph of nickel nanoparticles synthesized at 638 K, showing approximately spherical morphology and limited agglomeration

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The field-dependent magnetization curves exhibited hysteretic behaviour, indicating a finite coercive field and remanent magnetization under the reported measurement conditions. The extracted magnetic parameters at 20 K and 300 K are summarized in Table 1. As shown in Table 1 Magnetic parameters of the synthesized nickel nanoparticles.

Table 1 Magnetic parameters of the synthesized nickel nanoparticles

Parameters	20K	300K
Saturation Magnetisation, M_s (emu g^{-1})	32.2	32.5
Remanent Magnetisaton, M_r (emu g^{-1})	11.2	2.48
Coercive Field (T)	0.0284	0.0039
Ramanence Ratio (M_s/M_r)	0.318	0.076
Normalised saturation Magnetisation	1.0000	0.923

Both saturation magnetization values are lower than the approximate bulk reference value of 55 emu g^{-1} cited in the original account. Such differences may be associated with nanoscale surface effects, surface-bound organic species, oxidation, defects, and the magnetic structure at particle surfaces. The present measurements do not independently identify the relative contribution of each mechanism.the remanent magnetization. The value decreased from 11.2 emu g^{-1} at 20 K to 2.48 emu g^{-1} at 300 K. Similarly, the coercive field decreased from 0.0284 T to 0.0039 T. These changes indicate that the nanoparticles retain substantially less magnetization after removal of the applied field at room temperature than at 20 K. The temperature-dependent coercivity may reflect thermally activated magnetization reversal, changes in effective magnetic anisotropy, and interparticle interactions. Establishing the dominant mechanism would require additional information, including complete hysteresis loops, measurement-field range, particle-size distribution, and measurements over a wider temperature range.

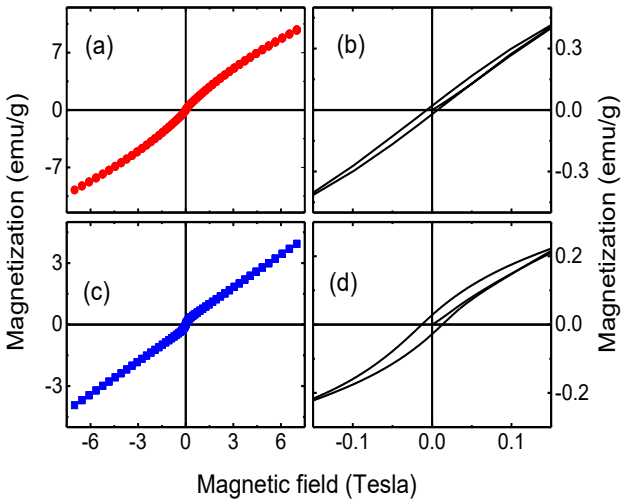


Figure 3 Field-dependent magnetization curves of nickel nanoparticles measured at (a) 20 K and (b) 300 K. The corresponding low-field regions should be shown separately in panels (c) and (d) to facilitate comparison of remanence and coercivity

The combined structural, morphological, and magnetic results provide a basis for relating the synthesis conditions to the measured properties of nickel nanoparticles. The fcc crystalline structure, approximately spherical particle morphology, and nanoscale dimensions were obtained following thermal decomposition at 638 K.The difference between the crystallite size and average particle diameter highlights the importance of distinguishing crystalline domains from physical particles. This distinction is relevant to magnetic measurements because surface contributions, interparticle interactions, and domain structure may affect the measured magnetization. The reduced saturation magnetization relative to the cited bulk reference and the decrease in coercivity at room temperature demonstrate that the magnetic response of the synthesized material is temperature dependent. Nevertheless, further measurements are required to establish a quantitative relationship between particle size, surface chemistry, magnetic

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anisotropy, and coercive behaviour. The results support the use of controlled thermal decomposition as a route for preparing nickel nanomaterials for further investigation in magnetic and catalytic applications. Actual application performance, however, would require dedicated testing rather than being inferred solely from structural and magnetic characterization.

Conclusion

Nickel nanoparticles were synthesized by thermal decomposition of nickel oleate in 1-octadecene and oleic acid at 638 K under a nitrogen atmosphere. X-ray diffraction revealed reflections consistent with the face-centred cubic phase of nickel, with an estimated lattice parameter of 0.350 nm and a Scherrer crystallite size of approximately 21 nm. FESEM analysis showed approximately spherical particles with an average diameter of about 28 nm, based on measurements of 100 particles. Magnetic measurements revealed saturation magnetization values of 35.2 emu g^{-1} at 20 K and 32.5 emu g^{-1} at 300 K. The remanent magnetization and coercive field also decreased substantially with increasing temperature. These observations demonstrate that the structural and magnetic characteristics of the synthesized nickel nanoparticles should be considered together when evaluating their potential applications. Further work involving quantitative particle-size distributions, surface-chemical analysis, oxidation-state characterization, and temperature-dependent magnetic measurements would provide a more complete understanding of the relationship between synthesis conditions and magnetic behaviour. Such studies would also help establish the reproducibility and suitability of the material for specific technological applications.

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