NiO Nanoparticle Synthesis Using a Triblock Copolymer: Enhanced Magnetization and High Specific Capacitance of Electrodes Prepared from the Powder

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ABSTRACT: Nickel oxide nanoparticles of diameter ~21 nm were prepared by a sol–gel method using the triblock copolymer poly(ethylene glycol)-b-(propylene glycol)-b-(ethylene glycol). X-ray photoelectron spectroscopy analysis showed the presence of Ni2+ and Ni3+ ions in the material. The electrical conductivity of this material was due to small polaron hopping between Ni2+ and Ni3+ sites. The magnetization shown by these nanoparticles was much higher than that reported in the literature. This is ascribed to the presence of Ni3+ ions with uncompensated spin moments. Spin-glass behavior was exhibited by the material at 10.7 K. The electrochemical characterization of electrodes comprising of these NiO nanoparticles using cyclic voltammetric measurements showed a specific capacitance value of 810 F/g, the highest reported for this material. These materials will thus form one of the useful multifunctional systems.

1. INTRODUCTION

Nanoparticles of nickel oxide have attracted the attention of the scientific community in recent years because of their properties that have been found to be suitable for applications such as catalysis, battery cathodes, gas sensors, electrochromic films, and chemical sensors.1–16 Several interesting basic studies have been reported to understand the mechanisms involved in the appearance of ferromagnetic (FM)-like behavior in antiferromagnetic (AFM) nanoparticles such as those of NiO. Using extended X-ray absorption fine structure technique, it was shown that magnetization in this system arose due to the presence of vacancies over the particle volume, which was described within a core−shell model. By extending the concept of vacancy-induced magnetism of nonmagnetic materials to an AFM system such as NiO, exchange bias property could be explained.17 The magnetic properties of NiO nanoparticles of different particle sizes showed that the blocking temperature increased as a function of diameter whereas the magnetic anisotropy decreased with an increase in the particle size. The uncompensated spins on the nanoparticle surfaces were found to be randomly distributed.18 For NiO nanoparticles with diameters less than ~12 nm, saturation and remanent magnetization and coercivity increased as the particle size was reduced.19 NiO nanoparticles synthesized using a simple thermal decomposition route exhibited FM properties including spin-glass behavior. The significance of surface states at the FM particle shell was brought out in such studies.20 NiO nanoparticles prepared using the ball-milling process showed FM behavior. High-temperature measurements exhibited a magnetic phase transition at around 780 K, which was due to the induced FM phase.21 A significant development has been the work related to the use of NiO as electrochemical capacitor electrodes as a replacement for RuO2 whose high cost limits its wide applications.22–30 Various preparation techniques have been reported for making NiO to study its electrochemical capacitance performance. The examples are NiO with an ordered mesoporous structure using mesoporous silica as a template31 or using a supramolecular templating mechanism;32 NiO/Ni(OH)2 nanoplatelets;33 mesoporous NiO with various hierarchical nanostructures;34 NiO nanoparticles using carbon black powder;35 capsule-like NiO nanoparticles;36 and porous hollow spheres of NiO nanosheets.37 A pluronic triblock copolymer has been used earlier to prepare nanocrystalline MgO with a large surface area.38 Similar studies have been reported in the synthesis of ZnO nanostructures.39 We have explored the synthesis of NiO nanoparticles using a triblock copolymer. These materials exhibited a FM behavior with high values of magnetization and showed excellent electrochemical characteristics as electrodes of supercapacitors. These properties were attributed due to the presence of both Ni2+ and Ni3+ ions on the surfaces of NiO nanoparticles synthesized using P123 triblock copolymer. The details are reported in this paper.

2. EXPERIMENTAL SECTION

2.1. Synthesis. The precursors used were poly(ethylene oxide)-b-(propylene oxide)-b-(ethylene oxide) triblock copoly-
mer P123, ethanol, and nickel acetate. A solution was prepared by dissolving 1 g of P123 in 12.67 mL of ethanol and 2.5 g of nickel acetate. After stirring it for 10 min by adding 0.6 g of 0.5 N HCl, 1−2 g of (C2H5)3PO was added, and the resultant mixture was stirred in a sealed bottle for 20 h. The sol was gelled in a Petri dish at 300 K in air for 5 days and subsequently dried at 353 K for 7 days in an oven. The sample was then heat-treated at 673 K for 6 h. The triblock copolymer template produced nucleation sites for NiO particles, which then grew during the subsequent heat treatment, and the organic entities were removed. The NiO nanoparticles with nanopores within them helped create the point defect Ni3+ on the surfaces of the particles. Powders obtained after crushing the product were used for characterization purposes (Figure 1).

2.2. Characterization. An X-ray diffraction pattern of the powders synthesized was recorded on a Bruker D8 XRD SWAX diffractometer using Cu Kα radiation. The microstructure was studied using a JEOL 2010 high-resolution transmission electron microscope. X-ray photoelectron spectra were recorded using a spectrometer supplied by Omicron Nano Technology with the serial no. 0571 using the Al Kα radiation source under 15 kV voltage and 5 mA current. Electrical resistivity measurements were taken on pellets made by compacting NiO powders prepared as described above by applying a load of 5 ton. Silver paint electrodes (supplied by Acheson Colloiden B.V., The Netherlands) were applied on both faces of the pellets. A Keithley-617 electrometer was used to study the resistivity variation as a function of temperature. Magnetization measurements in the temperature range 2−300 K were recorded using a magnetic property measurement system superconducting quantum interference device magnetometer (Quantum Design USA).

Electrochemical characterization was carried out using a three-electrode system. The working electrode was prepared by mixing 70% of active materials (in this case, NiO powder), 20% of acetylene black as conductive fillers, and 10% of polytetrafluoroethylene as a binder. The mixture was pasted on nickel foam (1 × 1 cm), which served as the current collector. The nickel foam was dried in an oven for 12 h at 353 K and then pressed to fabricate electrodes. A platinum wire was used as the counter electrode, and Ag/AgCl was used as the reference electrode. All measurements were recorded using 6 M KOH as the electrolyte. Cyclic voltammetric (CV) measurements were recorded on the above-mentioned system using a Neware BTS battery tester within a range of −0.2 to +0.3 V at the scan rates of 5, 10, 20, 30, 40, 50, and 70 mV/s.

3. RESULTS AND DISCUSSION

Figure 2 gives the X-ray diffraction pattern obtained from the NiO nanoparticles prepared in this work. The diffraction peaks have been indexed to the NiO lattice planes, as given in JCPDS no. 04-0835. Using the Debye−Scherrer equation, the crystallite size was estimated to be 20.9 nm. Figure 3a shows a transmission electron micrograph of the NiO nanoparticles. The average diameter of the particles is seen to be 21 nm, which is in satisfactory agreement with that estimated from the X-ray line-broadening analysis. A high-resolution transmission electron micrograph with clear evidence of lattice fringes for the planes (111) is shown in Figure 3b.

Figure 1. Synthesis of NiO nanoparticles using a triblock copolymer.

Figure 2. X-ray diffraction pattern obtained from NiO nanoparticles.

Figure 3. (a) Transmission electron micrograph of NiO nanoparticles. (b) HRTEM image of NiO nanoparticles.
nanopores within the NiO nanoparticles as pointed out earlier. The surfaces possess high energy content and thereby can sustain the generation of defects such as Ni$^{3+}$.

Figure 5 shows the variation in log resistivity ($\rho$) as a function of inverse temperature ($T$) of a pellet of NiO nanoparticles. The Ni$^{2+}$ and Ni$^{3+}$ sites give rise to a small polaron hopping mechanism of electrical conduction. According to this model, the DC resistivity $\rho$ is given by

$$\rho = \frac{kTR}{\nu_0^2C(1 - C)} \exp(2\alpha R) \exp\left(\frac{W}{kT}\right)$$  \hspace{1cm} (1)$$

where $k$ is the Boltzmann constant, $T$ is the temperature, $R$ is the average site separation, $\nu_0$ is the optical phonon frequency, $\epsilon$ is the electronic charge, $\alpha$ is the spatial decay parameter for the electronic wave function, which describes the localized state at each Ni site, $C$ is the ratio of Ni$^{2+}$/total Ni ion concentration, and $W$ is the activation energy. From the slope of the straight line representing the linear plot in Figure 5, the activation energy $W$ was found to be 0.23 eV. The resistivity data were fitted to eq 1, taking $R$, $\alpha$, and $\nu_0$ as fitting parameters. The extracted values of the latter are summarized in Table 1.

The values are reasonable. $R$ matches the intersite separation between Ni ions as calculated from the lattice constant of a NiO (rocksalt) structure.\(^{19}\) The electron localization parameter $\alpha$ and $\nu_0$ are also consistent with the theoretical model.\(^{18}\)

Figure 6a gives the variation in magnetization as a function of temperature in the range 2–300 K for a zero-field-cooled (ZFC) and a field-cooled (FC) sample by applying a field of 50 Oe. Here, the bifurcation temperature (above which all spins are unblocked and usually marks the highest blocking temperature of the ensemble) is above 300 K, and $T_B$ (a peak below the bifurcation temperature) cannot be measured exactly as the peak is very broad (169.5 K). At low temperatures, there is a sharp peak, which is designated by $T_B$ at 10.1 K. The small peak is due to the spin-glass behavior. For this, we have taken the AC magnetization measurement as a function of frequency in the temperature range 5–50 K. The experimental results are shown in Figure 6b, where variation in the real part of susceptibility is shown as a function of frequency. It can be seen that the curve shows a maximum, which shifted to higher temperature as the frequency is increased.

It can be seen from Figure 6b that the curve shows a maximum, which shifted to higher temperatures as the frequency is increased. Three models are used to analyze the results. The Neel–Arrhenius law should be applicable in the case of superparamagnetic systems for which the relaxation time $\tau$ is given by\(^{49}\)

$$\tau = \tau_0 e^{(E_i/k_BT_B)}$$  \hspace{1cm} (2)$$

where $E_i$ is the isotropy energy barrier, $k_B$ is the Boltzmann constant, $T_B$ is the peak temperature, and $\tau_0$ depends on the gyromagnetic precession time. Usually for superparamagnets, $\tau_0$ lies in the range of $10^{-10}$ to $10^{-13}$ s.\(^{49}\) However, in the present system, $\tau_0$ is extracted to be on the order of $10^{-26}$ s. This is unphysical; hence, the possibility of the presence of superparamagnets can be ruled out.

For a spin-glass system, the Vogel–Fulcher law\(^{49}\) should be applicable. For this, the value of $\tau$ is given by

$$\tau = \tau_0 e^{(E_i/k_B(T - T_B))}$$  \hspace{1cm} (3)$$

The experimental data were fitted satisfactorily to eq 3 as shown in Figure 7a. The large value of $\tau_0 \approx 10^{-9}$ to $10^{-8}$ s is expected from interacting magnetic spin clusters. The Vogel–Fulcher law is the modification of the Neel–Arrhenius law. As mentioned earlier, the Vogel–Fulcher law is used for both cluster-spin glasses and spin glasses. The power law assumes

![Figure 4. (a) XPS spectrum of O obtained from NiO nanoparticles. (b) XPS spectrum of Ni obtained from NiO nanoparticles.](image)

![Figure 5. DC resistivity variation as a function of inverse temperature for NiO nanoparticles.](image)

| composition      | $C$  | $W$ (eV) | $R$ (Å) | $\alpha$ (Å$^{-1}$) | $\nu_0$ (s$^{-1}$) |
|------------------|------|----------|---------|---------------------|--------------------|
| NiO nanoparticles| 0.56 | 0.23     | 4.3     | 1.7                 | $1.3 \times 10^{13}$ |

$^a$ $R$, $\alpha$, and $\nu_0$ were taken as the fitting parameters.
the existence of true equilibrium phase transition. According to the power law

\[ \tau = \tau_0 \left( \frac{T_p}{T_g} - 1 \right)^{-\nu} \]  

(4)

\( T_g \) is the freezing temperature, \( z \nu \) is a critical exponent, and \( \tau_0 \approx 10^{-10} \) to \( 10^{-13} \) is for the spin-glass system. The fitting of data to eq 4 is shown in Figure 7b.

It has been shown that 5 \( \leq z \nu \leq 11 \) for the spin-glass system and 1.2 \( \leq z \nu \leq 2 \) for regular ferromagnets.49

The values obtained for the different parameters using different models mentioned above are summarized in Table 2.

As stated earlier from the Neel–Arrhenius law, the value of \( \tau_0 \) obtained is unphysical, so the observed susceptibility variation is not caused by noninteracting superparamagnets. \( \tau_0 \) obtained from the Vogel–Fulcher law indicates the presence of conventional spin glass. The value of \( \tau_0 \) obtained from power law is about 4 orders of magnitude smaller than that extracted from the Vogel–Fulcher law. The difference between \( T_0 \) and \( T_g \) is very small, \( \sim 1.1 \) K.

Also, the initial frequency shift of \( T_p \) from the frequency dependence of the peak temperature was calculated using

\[ \delta T_p = \frac{\Delta T_p}{T_p \Delta \log_{10} \omega} \]  

(5)

where \( \delta T_p \) is the relative change in the freezing temperature and \( \Delta T_p \) is the total change in \( T_p \) in the frequency interval, \( \Delta \log_{10} \omega \). In the present system, \( \delta T_p = 0.04 \). For canonical spin glasses, it lies between 0.0045 and 0.06, and for superparamagnets, it is larger than 0.1.47 Thus, the studies on AC magnetization and magnetic relaxation confirmed the presence of spin-glass-like behavior in our system. The sample exhibited spin-glass behavior at low temperatures, with the freezing point at 10.7 K.

Figure 8a shows the magnetization–magnetic-field hysteresis curves obtained for the NiO nanoparticles at different temperatures under both FC and ZFC conditions. The magnetization obtained is much higher than that reported earlier in the case of NiO nanoparticles.47,48 Magnified views of \( M-H \) curves for low values of \( H \) are shown in Figure 8b. For FC \( M-H \) measurements, the sample that was cooled down to respective temperatures under 3 T magnetic fields exhibited an exchange bias effect, indicating the presence of unidirectional anisotropy.

Such an effect has been explained to arise because of the magnetic interaction at the interface between a FM and an AFM phase.37,49 At lower temperatures, exchange interaction is maximum, but at higher temperatures, the magnetic moments both in core and in shell are free to reverse with applied magnetic field, and thus, there is no shift in the hysteresis loop.

### Table 2. Three Models Used To Analyze the Variation in Magnetization as a Function of Temperature

|                  | Neel–Arrhenius law | Vogel–Fulcher law | power law |
|------------------|--------------------|-------------------|-----------|
| \( \tau_0 \) (s) | \( 1.4 \times 10^{-26} \) | \( 3.3587 \times 10^{-8} \) | \( 7.12 \times 10^{-12} \) |
| \( E_a/k_B \) (K) | 644.39             | 11.21             |           |
| \( T_0 \) (K)    | 9.59               |                   |           |
| \( T_g \) (K)    | 10.7               |                   |           |
| \( z \nu \)      | 7                  |                   |           |
In the present system of NiO nanoparticles synthesized using the P123 triblock copolymer, the exchange bias effect was much larger than those reported earlier.\(^{37,48}\) We ascribe such high values of magnetization observed to the presence of Ni\(^{3+}\) ions, which contribute to uncompensated magnetic spins in the otherwise AFM NiO system. This in addition to the oxygen vacancies at the nanoparticle surfaces gives rise to a high value of magnetization in the system.

### 3.1. CV Measurements

The specific capacitance was calculated using the following formula\(^{37}\)

\[
C = \frac{1}{mv(V_s - V_c)} \int_{V_c}^{V_s} I(V) \, dV
\]

where \(C\) is the specific capacitance, \(m\) is the mass of NiO powder, \(v\) is the scan rate (V/s), and \(I(V)\) is the anodic or cathodic current.

The specific capacitance of this material was calculated to be 810, 572, 522, 402, 337, 328, and 280 F/g at scan rates 5, 10, 20, 30, 40, 50, and 70 mV/s, respectively, as shown in Figure 9.

The electrochemical irreversibility exists due to the unsymmetrical cathodic and anodic sweeps.

Nowadays, transition metal oxides are promising materials that are used in electrochemical applications. Hydrous ruthenium oxide has exhibited a high specific capacitance value (720 F/g),\(^{30}\) but its high cost and toxicity remain as the problem. Therefore, it can be substituted by transition metal elements, which are cheaper and nontoxic. So far, a variety of NiO nanostructures such as nanoparticles, nanotubes, nanoflakes, and nanowhiskers were used as electrodes, and the maximum specific capacitance was found to be 580 F/g at a scan rate of 5 mV/s.\(^{33}\) The other reported capacitance of the electrodes made of nanostructured NiO materials had values in the range of 128−556 F/g.\(^{32,37}\) Our material gave the highest specific capacitance (810 F/g at 5 mV/s scan rate). It is believed that such an increase in capacitance is due to the enhancement of Ni\(^{2+}\) to Ni\(^{3+}\) redox reaction because of the presence of Ni\(^{3+}\) ions in our material as a result of the synthesis method adopted in this investigation. These results show that the NiO nanostructure prepared in the present work can be considered to be a promising candidate for the supercapacitor applications as it exhibits excellent electrode material characteristics.

### 4. CONCLUSIONS

Nickel oxide nanoparticles of diameter \(\sim 21\) nm were synthesized by a sol–gel method using the triblock copolymer poly(ethylene glycol)−poly(propylene glycol)−poly(ethylene glycol). XPS analysis confirmed the presence of Ni\(^{2+}\) and Ni\(^{3+}\) ions in the material. The electrical conductivity of the material was found to be due to small polaron hopping between Ni\(^{2+}\) and Ni\(^{3+}\) sites. The magnetization exhibited by these nanoparticles was much higher than those reported in the literature for this system. This is ascribed to the presence of Ni\(^{3+}\) ions with uncompensated spin moments. Spin-glass behavior was shown by the particles at 10.7 K. Electrochemical characterization of electrodes comprising of these NiO nanoparticles using CV measurements gave a specific capacitance value of 810 F/g, the highest reported so far. These materials will form one of the useful multifunctional systems.

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**Figure 8.** (a) \(M−H\) hysteresis curves for NiO nanoparticles at different temperatures. (b) Magnified view of \(M−H\) curves for low values of \(H\).
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Notes
The authors declare no competing financial interest.

ACKNOWLEDGMENTS
S.C. and M.M. acknowledge the award of INSPIRE Fellowship by the Department of Science and Technology, New Delhi. R.M. thanks the Council of Scientific and Industrial Research, New Delhi, for the award of a Research Associateship. D.C. thanks the Department of Science and Technology, New Delhi, for the award of SERB Distinguished Fellowship and the Indian National Science Academy, New Delhi, for giving him an Emeritus Scientist position.

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