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# Nanometric study of Nickel Oxide Prepared by Sol Gel Process

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**Abstract**.Nickel oxide nanopowder was synthesized by sol gel method using nickel nitrate as the starting material. Nickel oxide nanoparticles with a grain size of 15-90 nm have been studied by; small angle neutron scattering; scanning electron microscopy; and vibrating sample magnetometry. A combination of Ferro and paramagnetic behaviour of the particles after calcination at 800 °C is observed while for powder calcined at 400 °C, soft magnetic character with saturation is seen. The system of nanoparticles ofNiO embedded in a silica matrix is also studied for the structural change. Weak magnetic ordering is observed in this case with the likely-hood of particles being evenly distributed in the silica.

## INTRODUCTION

Nanostructure materials have been studied extensively for technological applications as well as in fundamental science to prepare new functional materials [1]. In Industries such as agriculture, food, iron and steel, mining, pharmaceutical, chemical manufacturing etc., a large concern is the enormous amount of aqueous waste that is produced. The waste water from these is adversely contaminated by organic pollutants, bacteria, microorganisms and also the industrial effluent is contaminated with heavy metals or petroleum derivatives. Hence it is required to develop new water treatment technologies in order to ensure a clean supply of drinking water and reduce water pollution. The objectives of the present study is synthesis and structural investigation of magnetic porous materials that could find applications in waste water treatment, removal of toxic materials from medical/industrial wastesand other such applications [2].

Nickel oxide [3]has a wide range of applicationsin the manufacture of various composites, magnetic materials etc. To tailor its various applications the Magnetic nanocomposites may be partially embedded in a porous network of secondary material [4]. Embedding will distribute magnetically active centers in porous matrix that will trap magnetic impurities and nonmagnetic impurities will be filtered.Such magnetic porous materials are well suited for applications in water purification, bacteria filtration, gas separation, molecular filtration, adsorbent etc.. Understanding the pore morphology, surface area and their connectivity are crucial for technological applications of porous materials [5].Transport properties in such materials depend on interconnection as well as pore morphology.Nano-structures of magnetic nickel oxide nanoparticles and embedded particles in mesoporous silica have been studied by small angle x-rays scattering (SAXS), small angle neutron scattering (SANS)[6] and vibrating sample magnetometer (VSM).

## EXPERIMENTAL METHODS

Magnetic Nanostructured Nickel oxide (NiO) was synthesized by sol-gel method using Nickel nitrate ($Ni(NO_3)_2.6H_2O$) and citric acid ($C_6H_8O_7.H_2O$) as starting material [7]. Nickel nitrate solution was dripped in the solution of citric acid stirring vigorously. The solution was heated at temperature 70 °C for 12 hoursand the obtained product was washed with distilled water and alcohol. The translucent gel was dried at 110 $^0$C for 24 hours. The green powder formed was calcined at three different temperatures, i.e., 400 $^0$C, 600 $^0$C and 800 $^0$C to obtain black NiO particle. Nanoparticles calcined at 400 °C was embedded in $SiO_2$at temperature of 1000 °C for further investigation. Structural and magnetic properties of nanostructures were characterized by X-ray

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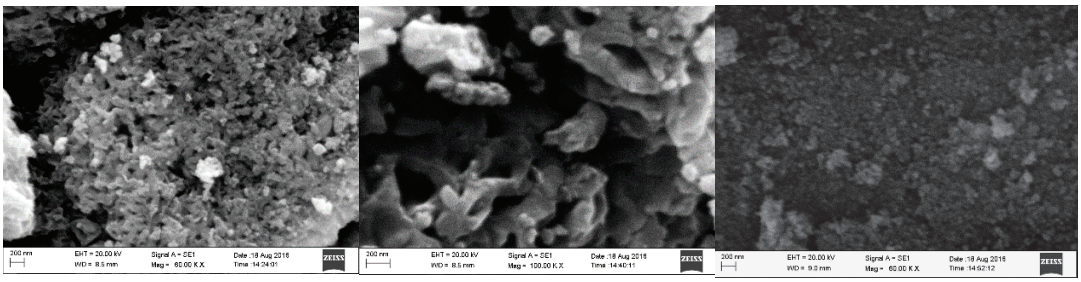
diffraction (XRD)Rigaku X-ray diffractometer with incident CuKα (λ = 1.5418 Å) in the scattering range $15^0$ to $80^0$. Small Angle Scattering of X-rays (SAXS) and Neutrons (SANS) in the Q ranges 0.1 nm-1 to 2 $nm^{-1}$ and 0.003 to 0.173 $nm^{-1}$ respectively and Quantum Design made 9 Tesla PPMS based vibrating sample magnetometer (VSM) technique at UGC DAE centre Trombay. The JEOL (15kv) SEM instrument used to study the surface morphology as a function of sintering temperature.

## RESULTS AND DISCUSSIONS

Nickel oxide phases were confirmed by X-ray diffraction pattern with presence of few impurity reflections at lower values of scattering angle. The heights of some of the NiO peaks are found to decrease with increase in calcination temperature. This would be expected as the $SiO_2$ network begins to disrupt and some of the NiO crystal planes get affected. The average particle size determined from the Scherer formula using full width at half maximum at various diffraction peaks was 20 nm for powder calcined at 400 $^0$C which increased to 22 nm for the powder calcination at 600°C.SEM micrographs revealed that the particles had elongated shape and are agglomerated.The agglomeration of Nano crystalline particles could be attributed to their extremely small dimensions with high surface energy during the polycondensation and drying steps of the sol gel method. Particles with sizes ~ 20 nm were observed from SEM for particles calcined at 400 °C as shown in fig.1. The particle size was found to increase to 50 nm when the calcination temperature was increased to 600 °C indicating a crucial role of calcination temperature on the morphological structure of NiO particle. The SEM micrograph of nanoparticles embedded in SiO2 matrix is shown in fig.1(c).



(a) (b) (c)

**FIGURE1**. SEM micrographs of nickel oxide nanoparticles calcined at (a) 400 °C (b) NiO 600°C (c) NiO calcined at 400 °C and embedded in $SiO_2$

Small-angle neutron scattering experiments have been performed using a double crystal based medium resolution (MSANS) instrument at Guide Tube Laboratory Dhruva reactor Trombay [7]. In SANS the scattering originates due to density fluctuations or structural inhomogeneities in the material. For an isotropic system of polydisperse particles the variation of scattering intensity with Q is given by:

$$I(Q) = \frac{N_p(\rho_p - \rho_m)^2 V_p^2}{V_{sample}} P(Q)S(Q) \qquad 1$$

where P(Q) is the single particle formfactor and S(Q) is the inter-particle structure factor. For nickel oxide nanoparticles neutron scattering contrast is significant.The SANS and SAXS profile of magnetic nickel oxide nanoparticles and the particles embedded in mesoporous silica are shown in fig.2. The SANS and SAXS data recorded by the instrument were corrected for background, transmission and resolution broadening. The analysis of SANS data is carried out using SASFIT programme.It has been assumed that the scattered intensity is obtained from polydispersed spherical shaped particles.

The nickel oxide powdercalcined at 400°C, the SANS pattern shows broadly two broad maxima with two length scales. For the sample calcined at 800 ° C, these maxima shift to higher Q values with an increase in intensity.The SANS data for the NiO particles embedded in $SiO_2$ reveal scattering from inhomogeneities at three different length scales. The associated length scale obtained for this sample are ~590 nm, 85nm, 17 nm and an aggregation size of 59 nm. SAXS data was fitted asumming spherical shape for the sample calcined at 400 °C the particle size obtained from the fit is 19 nm. The silica compacts with embedded magnetic NiO nanoparticles have useful applications in filtration and waste water treatment.

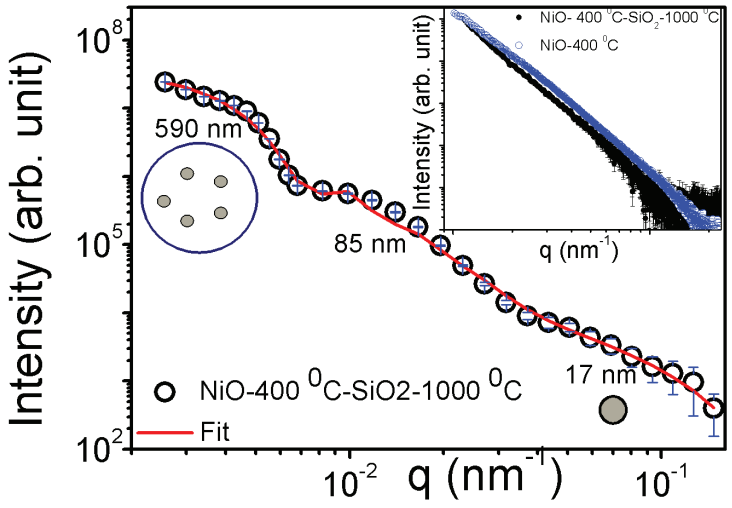



**FIGURE 2**.SANS data on NiO particle embedded in SiO2 matrix (Insert SAXS data on NiO particlescalcined at 400 °C and embedded in $SiO_2$matrix).

Magnetic properties were measured by the Vibrating Sample Magnetometer at 300K and 3K. Magnetization loops obtained at both temperatures for NiOcalcined at 400 °C in figure 3(a) show soft ferromagnetic character with small hysteresis (Hc~ 155 Oe at 3K and 100 Oe at 300K). Whereas a combination of paramagnetic plus ferromagnetic behavior is seen for the sample calcined at 800 °C with much less coercivities (Hc ~ 80 Oe at 3K and < 20 Oe at 300K). Magnetization in the 400°C calcinedsample show complete saturation with saturation magnetization at 9T, Ms~ 34.7 emu/gm at 3K and 35.6 emu/g at 300 K, .whereas it has sharply fallen to Ms ~ 1,5 emu/g at 3K and 1.39 emu/g at 300 K. For the nanoparticles embedded in a silica matrix, the magnetization exhibits a paramagnetic like behavior with weak ordering at temperatures below 5K and it shows PM like MH loop with hysteresis of Hc ~ 100Oe with a Ms at 9T and 3K ~ 0.8 emu/g. This clear indicates that the NiO nanoparticles are evenly distributed in the $SiO_2$ matrix and the have very weak magnetic order seen for T< 5K which may may be due to agglomeration of some particles.Temperature dependence of nagnetization of nickel oxide particles at zero field cooled and field cooled conditions at magnetic field of 250 Oe were studied.

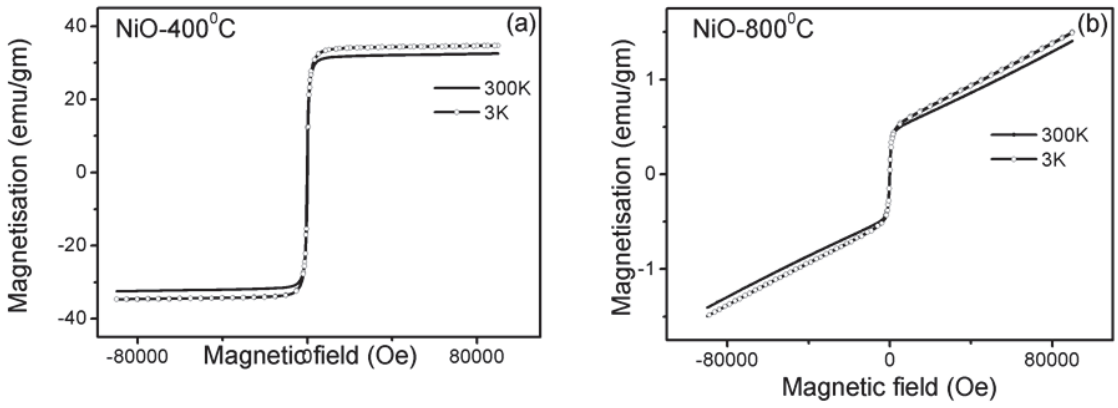



**FIGURE 3**. Magnetization vs applied filed at 3K and 300K for NiO nanoparticles sintered at (a) 400 $^0$C (b) 800 $^0$C

## CONCLUSIONS

NiO nanoparticles have been synthesized by the sol gel method and studied for their structural and magnetic properties by XRD, SANS, SAXS, SEM and VSM. The particle size was found to increase with calcined temperatures indicating a crucial role of calcination temperature on the morphological structure ofNiO particles. Two different sizes obtained from SANS study on NiO nanoparticles calcined at temperatures of 400 °C, 600°C and 800 $^{0}$C. For particles embedded inSiO2 matrix theassociated length scales obtained are ~590 nm, 85nm, 17 nm and an aggregation size of 59 nm. For calcination at 400 C, the samples have soft ferromagnetic character while the ones sintered at 800 C show both ferro and paramagnetic behavior. When the NiO particles are embedded in a silica matrix, theyexhibit near paramagnetic behavior with very weak magnetic order for T < 5K thereby indicating that NiO nanoparticles are evenly embedded in SiO2 matrixThe silica compacts with embedded magnetic NiO nanoparticles may have useful applications in filtration and waste water treatment.

## ACKNOWLEDGMENT

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