One pot facile synthesis of nanosized Nickel oxide by direct precipitation method

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ABSTRACT Nanosized nickel oxide has been synthesized by precipitation method and characterized by using XRD (X-ray diffraction), TGA/DTA, TEM (transmission electron microscopy) and Magnetic Measurements techniques. XRD studies show that nickel oxide was formed as NiO and it has octahedral structure. Magnetic measurements showed nickel oxide has two unpaired electron and is paramagnetic in nature. The particle size of the synthesized iron oxide was determined by TEM. TEM images show that the size of particles of NiO varied from 28nm to 52 nm which is in good agreement of the theoretically predicted size of nanomaterials. This method is convenient, easy and effective in comparison to the known methods of the synthesis of nanomaterials like thermal decomposition of precursors, co-implantation of metal and oxygen ions and ultrasonic spray pyrolysis.

Key words: Nanomaterial, nickel oxide, TEM, metal oxides, XRD analysis

1. Introduction:
Transition metal oxides have many applications as catalyst [1-5], sensors [6-9], superconductors [10-11] and adsorbents [12-13]. NiO has a variety of specialized applications and generally applications distinguish between "chemical", which is relatively pure material for specialty applications, and "metallurgical grade", which is mainly used for the production of alloys. It is used in the ceramic industry to make frits, ferrites, and porcelain glazes. The sintered oxide is used to produce nickel steel alloys. NiO was also a component in the nickel-iron battery, also known as the Edison Battery, and is a component in fuel cells. It is the precursor to many nickel salts, for use as specialty chemicals and catalysts. More recently, NiO was used to make the NiCd rechargeable batteries found in many electronic devices until the development of the environmentally superior Lithium Ion battery [14]. Nickel oxide has many catalytic applications as Nickel – Uranium oxide catalyst has been used for the hydrogenation of carbon dioxide to methane [15]. Potassium/calcium/nickel oxides has been observed as a good catalyst for the oxidative coupling of methane [16]. Mixed nickel–manganese oxides with an ilmenite and spinel structure have been investigated in order to elucidate the effect of the crystal structure type and cation coordination on the catalytic performance in the reactions of complete oxidation of ethyl acetate, benzene and carbon monoxide. Nickel–manganese oxides with an ilmenite-type structure are perspective catalysts for catalytic neutralization of exhaust gases from organic compounds [17]. Nickel–manganese mixed oxides NiMn₃O₄ spinel was used for the partial conversion of methane [18]. As nickel oxide has wide applications in industry and catalyst for reactions, an attempt has been made to synthesize nickel oxide nanoparticles by simple aqueous precipitation using ammonia as precipitating agent. This method involves a simple, cheap and one step process for synthesis of NiO nanoparticles. The obtained particles of NiO have size from 28-50nm. The synthesized nanoparticles were characterized by XRD, TGA, Magnetic susceptibility, SEM and TEM

2. Methods and materials
2.1 Chemicals:
All chemicals used in the experiment are analytic reagent grade. Nickel nitrate Ni (NO₃)₂ was purchased from Merck, India. Ammonium hydroxide (liquor ammonia) was purchased from SRL. Deionized water was used throughout the experiment.

2.2 Synthesis of iron oxide:
500 ml of 0.1M solution of Ni (NO₃)₂ was taken and aqueous ammonia was added drop wise with constant stirring until the pH of the solution reached to 10. The precipitates thus obtained were filtered by Buckner funnel and was washed several times with distilled water. The precipitates were dried in oven at 70°C for 24 hrs and were calcined at 600°C in a muffle furnace for 5 hrs. Obtained material was ground and sieved through 100 mesh size sieve.
2.3 Equipments:

The powder X-ray diffraction (XRD) was performed using X-ray diffractometer system Philips PW 11/90, with CuKα (λ = 1.5405 Å) radiation. Magnetic measurements were done using vibrating sample Magnetometer Model 155. TGA/DTA were done between a temperature range of 10-1000°C under N2 atmosphere using . The transmission electron microscopy (TEM) was performed with Hitachi H7500. SEM studies were carried out to find out the morphology of the synthesized oxide using scanning Electron Microscope Quanta 200 FEG (FEI Netherlands).

Results and discussions:

3.1. X-ray studies:

X-ray diffraction of synthesized oxide is shown in Figure (1). X-ray diffraction pattern of pure nickel oxide indicated that nickel oxide in the form of NiO [Fig- 1]. In X-ray diffraction, some prominent peaks were considered and corresponding d-values were compared with the standard [JCPDS file No. 05-661] [Table-1]. X-ray diffraction shows that metal oxide is pure NiO having octahedral structure.

3.2 Magnetic measurements:

The magnetic moment for nickel oxide is found to be 2.691 B.M. This value of magnetic moment supports the fact that the formed nickel oxide is in the form of NiO with actual magnetic moment 2.828 B.M. This indicates that 2 unpaired electron is present in NiO. Thus the oxide formed is paramagnetic in nature [Table 2].

3.3 TGA/ DTA studies:

From TGA curve we observed that nickel oxides show stable weight loss above 500°C [Fig.2]. It simply indicates that for the formation of nickel oxide temperature above 500°C is required.

3.4 SEM and TEM studies

SEM and TEM studies were performed to find out the morphology and exact particle size of synthesized NiO. SEM and TEM shows that the NiO nanoparticles are octahedral (Fig. 3, Fig4) and having particle size in the range of 28 – 50 nm which is in good agreement of nanosized particles [Table 3].

4. Conclusion:

NiO nanoparticles with octahedral structure are synthesized successfully by aqueous precipitation method. From TEM study, it is found that particles are with average size of 28-50nm. Magnetic measurements shows that NiO has two unpaired electron and hence paramagnetic in nature. This method is advantageous over the existing methods of synthesis of nanoparticles because other methods require specialized instrumentation, highly skilled labour, expensive materials and methods. Therefore, the proposed precipitation method is very promising and may have extensive applications.

Table 1

<table>
<thead>
<tr>
<th>S.No.</th>
<th>d=λ/2Sinθ (Observed)</th>
<th>d=λ/2Sinθ (Reported)</th>
<th>I/I₀x100% Observed</th>
<th>I/I₀x100% Reported</th>
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<tr>
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<td>2.41228</td>
<td>57.0568</td>
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<tr>
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<td>1.20443</td>
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Table 2

OBSERVATIONS ON MAGNETIC SUSCEPTIBILITIES OF SYNTHESIZED NICKEL OXIDE

Experimental Conditions:
Applied Magnetic field= 5.0 Kilo gauss
Temperature = 296K

<table>
<thead>
<tr>
<th>Sr.No</th>
<th>Name of Oxide</th>
<th>R=μ₁-μ₂ (e.m.u)</th>
<th>W=w₁-w₂ (gm)</th>
<th>μ_calculated = μ₁-μ₂</th>
<th>μ_observed (B.M.)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>Nickel Oxide</td>
<td>1.0635 x 10⁻²</td>
<td>0.05257</td>
<td>2.69</td>
<td>2.82</td>
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</table>

μ_calculated = \sqrt{n(n+2)} and μ_observed = \frac{2.84\sqrt{RTM}}{HW}

Where
n = no. of unpaired electron.
R = Magnetic Moment in B.M.
T = absolute Temperature.
M = Molecular weight
H = Applied Magnetic Field
W = Weight of Sample.

TABLE-3

PRACTICAL SIZE OF SYNTHESIZED NICKEL OXIDE AT DIFFERENT SCALES

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Scale (20nm)</th>
<th>Scale (50nm)</th>
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<tr>
<td>2</td>
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<td>8</td>
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<td>35</td>
</tr>
<tr>
<td>Range</td>
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<td>30nm to 52nm</td>
</tr>
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</table>
Figure 2: TGA/DTA curve of nickel oxide heated at 70° C

Figure 3: SEM micrographs of nickel Oxide particles
Figure 4: TEM micrographs of nickel oxide particles

References:
5. Simona B., Antonella G. and Vittorio R.(2003), Preparation of highly dispersed CuO catalysts on oxide supports for de-NO(x) reactions, Ultrasonic sonochemistry 10, 2 : 61-64.
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