

A Versatile Synthesis of Ni Nanoparticles by Solution Combustion Method

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ABSTRACT

We report here a simple solution combustion synthesis of Nickel oxide (NiO) nanoparticles by using glycine as a fuel and nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) as an oxidizer and its reduction to nickel nanoparticles by using hydrogen gas. The morphological structural properties of nanoparticles were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared (FT-IR) and UV-Visible spectroscopic technique. The solution combustion-synthesized Ni nanoparticles have a crystalline size of ~ 20 nm. The agglomeration of fine particles with particle size in the range of 30–50 nm is seen by SEM images. The FT-IR spectrum shows absorption band at ~ 466 cm^{-1} for Ni nanoparticles. UV-Vis. Spectroscopy shows absorption band for NiO at ~ 350 nm and Ni at 400 nm. Ni nanoparticles are used in different fields like CNT synthesis catalysis supercapacitors etc.

Keywords: Solution combustion, NiO, Glycine, Catalyst, CVD, H₂ gas, Nanoparticles

I. INTRODUCTION

From literature survey we learnt that, Nanoparticles of transition metals like Ni, Co, Fe etc. Have been studied by several scientists in the last few years. Nanoparticles of transition metals are getting continuous importance for various application such as catalysts (Patil K C 1997) super capacitors, gas sensors, biosensors, supercapacitors, Several researchers have prepared NiO by different method like sol-gel (C N R Rao 1963) surfactant-mediated synthesis (C N R Rao 1994) thermal decomposition (Rao K J and Ramesh P D 1995) solution combustion synthesis (S. Balmurgan and A.J. Linda Phillip 2016) and so on. However to the best and most commonly used laboratory synthesis is solution combustion method. In solution combustion method different combination of fuels and oxidizers are used oxidizers like metal nitrate, metal chloride metal sulphates etc., fuel like glycine, urea, citric acid, oxalic acid, glucose, sucrose, aniline.

Current report is solution combustion synthesis using nickel nitrate hexahydrate as an oxidizer and glycine as a fuel. The characterization study of prepared NiO and Ni nanoparticles was done by SEM, XRD, FTIR, UV-Vis spectroscopy.

II. MATERIALS AND METHODS:

Synthesis of Nickel oxide and Ni nanoparticles Very small particles of metals such as Ni, Co, Fe are known for their catalytic role in growth of CNT (Schwarz et al., 1995). Ni catalyst was prepared by thermal decomposition method. Nickel nitrate hexahydrate and glycine were mixed at fixed 1:1 molar ratio in 25ml distilled water and stir for 10 minutes. (Chatterjee et al., 2003). The solution was then kept in pre heated muffle furnace at 350°C at the flash point of glycine. The decomposition of glycine is highly exothermic and large amounts of ammonia and carbon dioxide are liberated and fine Nickel oxide was obtained. Nickel oxide produced in grind with hand paste to get fine powder. Nickel oxide obtained after grinding is blackish in colour and sonicated. Here NO_3^- in metal nitrate act as oxidizer and an organic compound that has carboxylate and/or amine (i.e. glycine) act as fuel. This is an autocatalytic and self-propagating reaction utilizing exothermic redox decomposition of fuel and oxidizer. Residual energy of combustion (reaction enthalpy) is used to crystallize the particle. The explosive gas blows off and material resulting into ultra-fine crystallite powder. The Nickel oxide was reduced in CVD furnace by H₂ at 600°C for 2 h yielding a very fine metal nanoparticle which was used as catalyst to grow CNTs by CVD (Turano et al., 2006). The prepared NiO and Ni

sonicated with ethyl alcohol for 20 min. further studied by using technique like SEM, XRD, FTIR, UV- Vis spectroscopy.

III. RESULTS AND DISCUSSIONS

SEM and XRD Study:

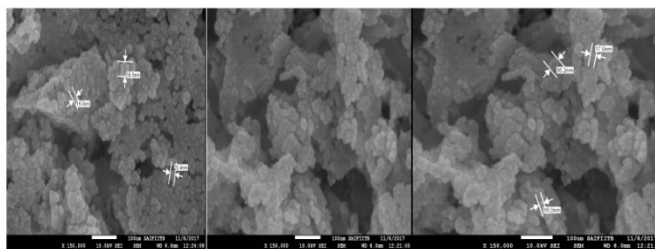


Figure 1. SEM images for Ni nanoparticles

$D = 0.94\lambda/\beta\cos\theta$ Where λ is wavelength of XRD radiation β is the full width at half maximum of the peak corresponding to the plane. θ is the angle obtained from 2θ value corresponding to the XRD pattern. Crystalline size obtained from the sharp peak at $2\theta = 44.77$ is 59.15 from the powdered XRD.

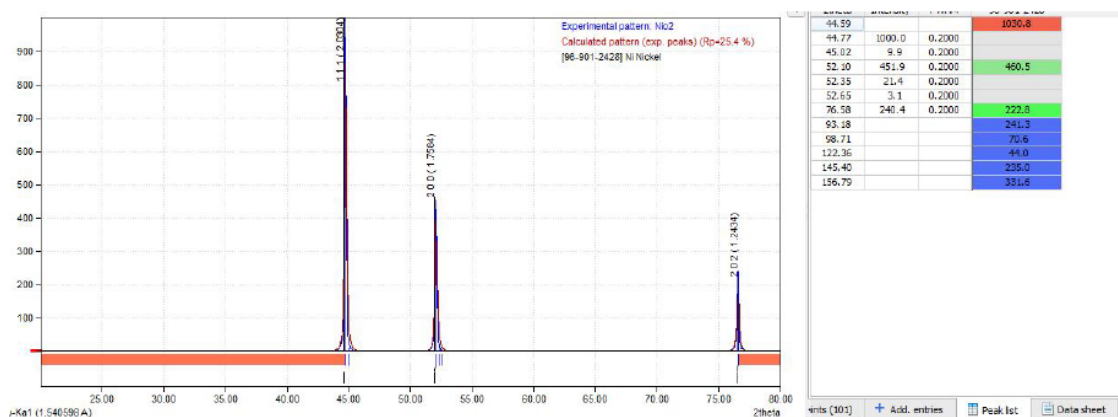


Figure 2. Ni nanoparticle XRD image

UV-Vis and FTIR Study

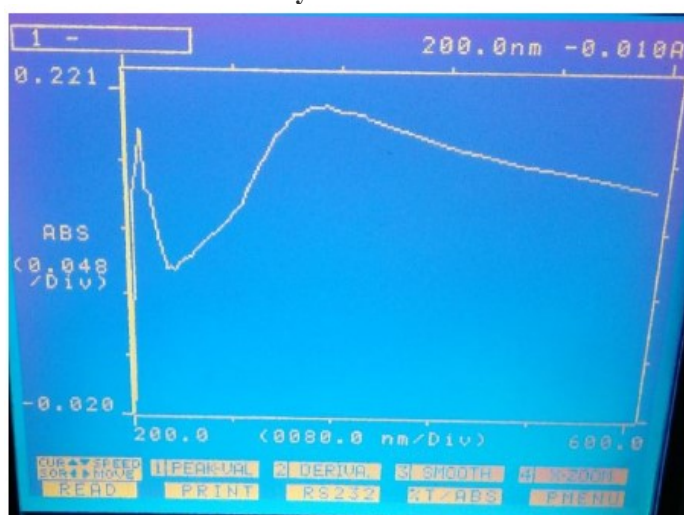


Figure 3.1. UV-Vis spectrum for NiO

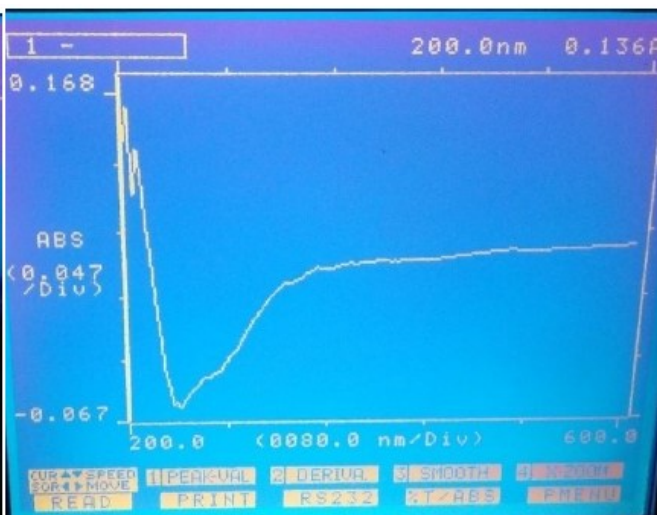


Figure 3.2. UV-Vis spectrum for Ni

The optical absorption behavior of the prepared NiO and Ni was examined in the UV-visible absorption region (200-600 nm), and their typical absorption spectrum is depicted in Figure 3.1 and Figure 3.2 for NiO two UV absorption peaks at ~209 and ~350 nm which indicate presence of NiO

from literature study. In Figure 3.2 maximum absorption observed at 400 nm indicates presence of Ni nanoparticle, further presence of Ni nanoparticles is supported by FT-IR. Peak for Ni nanoparticle is at around 466 cm⁻¹. Images taken during the synthesis of Ni nanoparticles from nickel nitrate hexahydrate and glycine are given in Figure 5.1, 5.2 and 5.3.

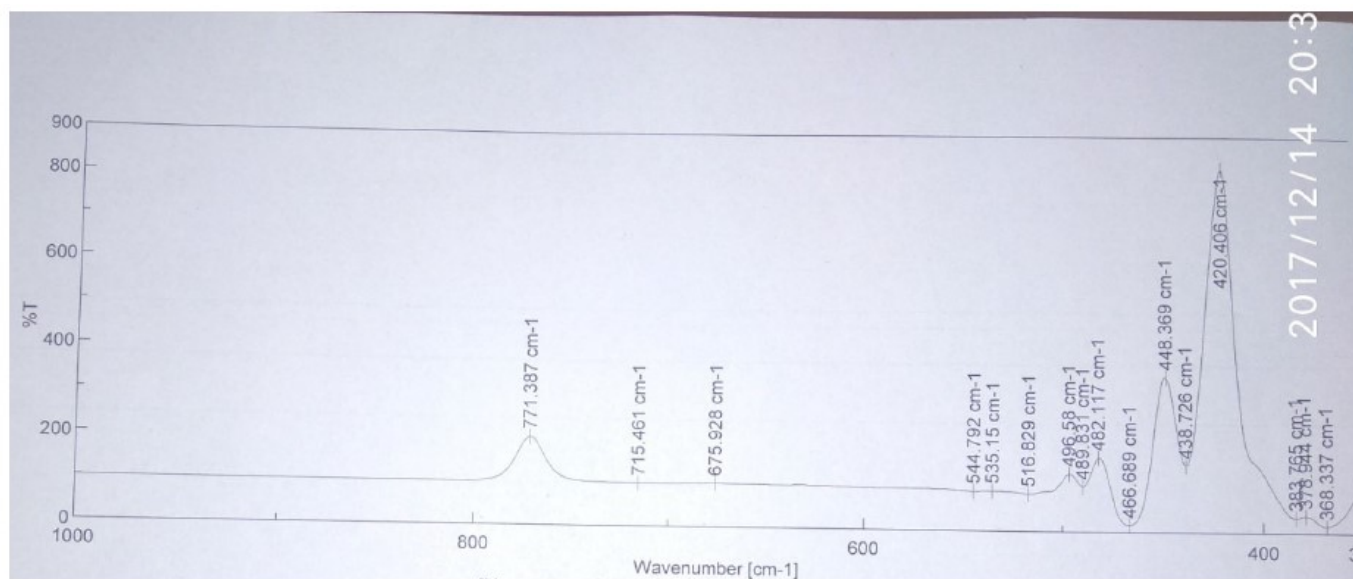


Figure 4. FT-IR spectrum for Ni nanoparticle

Images for NiO and Ni Synthesis



Figure 5.1. Flame at ignition point



Figure 5.2. NiO nanoparticles



Figure 5.3. Ni nanoparticles

IV. CONCLUSION

Ni nanoparticles were successfully synthesized by solution combustion method by using glycine as fuel with good yield 8-10%. The synthesized nanoparticle were highly pure and almost homogeneous in size ranging in between 20-40 nm. The prepared nanoparticle will be suitable for potential application in catalysis, supercapacitors, biosensors, electrochromic devices. In further investigation we are using synthesized Ni nano particles for the production of CNT by CVD and plant oil as precursor.

V. ACKNOWLEDGMENTS

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VI. REFERENCES

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