

Polymeric Nanocomposite Of Poly Vinylpyrrolidone- Nio : Synthesis And Its Characterisation

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ABSTRACT

In this research, nickel oxide (NiO) nanoparticles and Polyvinylpyrrolidone(PVP) of nickeloxide nanocomposites (PVP-NiO) with contents of PVP(50wt%)-NiO was prepared via in situ chemical method. Characterization of NiO nanoparticles and PVP-NiO nanocomposite was investigated by Fourier transforms infrared (FTIR), Scanning electron microscopy (SEM) and UV- visible spectrophotometer. The UV-visible spectrophotometry analyzing showed the strong absorption peak and nearly transparent nature of the particle at visible region. Moreover, the electrical conductivity of the nanocomposites were investigated at different temperature. FTIR spectra revealed to the formation of some interactions between the PVP and the Nickeloxide nanoparticles. The electrical conductivity of PVP–NiO nanocomposite was found to decrease due to the encapsulation of polymer on Nickel oxide nano particles. This study will be used as coating material, semiconductor devices and also used as sensor material for the detection of environmental organic pollutants in water and also traces of explosives.

Keywords: Nanoparticle, Composite materials, Polyvinylpyrrolidone, Electrical conductivity.

1. INTRODUCTION

Magnetic materials, alkaline battery cathodes, dye-sensitized solar cells, semiconductors, solid oxide fuel cells (SOFC), anti ferromagnetic layers, p-type transparent conducting films, electrochromic films, heterogeneous catalytic materials and gas sensors were made by using Nickel oxide.[T. Fukui(2000) - G. Wang (1997)]. Also, nanocrystalline Material of nanoNickel oxide showed superparamagnetism effect and also used for drug delivery and MRI agent [J.T. Richardson (1991)]. Nickel

oxide and its nano composite were prepared by various techniques like thermal decomposition [W. Wang (2002)-- X. Li (2006)], carbonyl method, sol-gel technique [L. Xiang (2002)], microwave pyrolysis [Y. Wang (1996)], solvothermal [K. Anandan (2011)], anodic arc plasma [Z. Wei (2009)], Sonochemical [S. Mohseni Meybodi (2012)], precipitation-calcination [X. Deng (2004)] and microemulsion [Y. Du (2012)]. Most of the techniques are having complicated steps and also the yield of the material is low. Wet chemical method is somewhat easier than other techniques and also getting the small sized nano particles. It gives comparatively better result in electrical and optical property of the material than any other techniques..

Poly vinylpyrrolidone (PVP), also commonly called polyvidone or povidone, is a polymer made from the monomer vinylpyrrolidone. It is flaky powder and absorbs water upto 40 % of its own weight and is water soluble and also in polar solvents and has excellent wetting properties and readily forms films. This makes it good as a coating or an additive to coatings. PVP is used in a wide variety of applications. PVP as an effective stabilizer and protecting agent can limit particle growth and prevent the particles from aggregation. PVP and metal nanoparticles formed a complex. by coordinate bonds between the surface sites of noble metal nanoparticles and N as well as O atoms of PVP, which resulted in a low concentration of free noble metal nanoparticles and a decrease in the electrode reduction potential of the metal ion, and then speeded up the reduction rate of metal ions as well as the consumption of metal atoms during particle growth process. As the concentration of PVP was excessive, it would restrain the diffusion of the metal nanoparticles. Therefore, the dispersion of the nanoparticles became more difficult and aggregation would be occurred again, leading to an increase in the metal nanoparticle size. The main objective is to study the structural and electrical properties of the polymer composite material of PVP and Nickel oxide.

2. EXPERIMENTAL

2.1 Materials

All the chemical reagents used in this study were of analytical grade and are used without further purification. Distilled water was used throughout the experiments.

2.2 Preparation of nanoNickel Oxide

The nanoNickel oxide was synthesized by following a modified chemical wet method. At 90°C, 100 ml of 2M NH₄OH was added in to 50 ml of 0.5M Ni(NO₃)₂·6H₂O and stirred for 24 h.. The product obtained after filtration was oven-dried overnight at 100 °C and the flakes were powdered using an agate mortar and pestle.

2.3 Preparation of PVP(Polyvinylpyrrolidone)- nanoNickel Oxide composite

PVP was dissolved by using magnetic stirrer for 3h and the polymer solution was left overnight in room temperature to remove the air bubbles trapped in the viscous solution Then suitable amount of nanoNickel oxide was dispersed in deionised water by 30-min ultrasonication (METAL POWER ANALYTICAL (I) PVT. LTD. Maharashtra, India). Ultrasonication was necessary to avoid agglomeration of powder and to achieve proper dispersion. Nano nickel oxide in water was mixed with polymer solution under agitation. The homogeneously mixed solution is immediately taken to deep freeze at -18°C. After 48 h of freezing the samples were freeze dried. The nanoNickel oxide- PVP composites were coded as nanoNiO-PVP 50 where number denotes the wt% of PVP.

2.4 Characterization methods

The synthesized PVP- nanoNickel oxide composite was characterized spectrophotometrically using Perkin Elmer UV-visible spectrometer. The morphology of the materials was analyzed by Field-Emission Scanning Eletron Microscopy (FE-SEM) using a HITACHI S600N scanning electron microscopy (HITACHI High Tech, Japan). Functional group confirmations were assessed by SHIMADZU IR-affinity FTIR spectrophotometer. Electrical properties of nanoNiO and PVP- nanoNickel oxide composite also were determined by using conductivity meter for various temperature.

3. RESULTS AND DISCUSSION

FT-IR studies

FTIR spectra of Fig 3.1 showed the pure nanoNickel oxide and appeared the predominant peaks at the wave number of 3632 cm⁻¹ which was due to the O-H vibration and was formed at higher frequency due to splitting of water molecules on the surface of Nickel oxide nanoparticles. The peak of C-H in plane bending modes

appeared at 1304 cm⁻¹ and the peaks at 414 cm⁻¹ to 590 cm⁻¹ corresponds to metallic stretch and it can be assigned to nickel oxide groups.

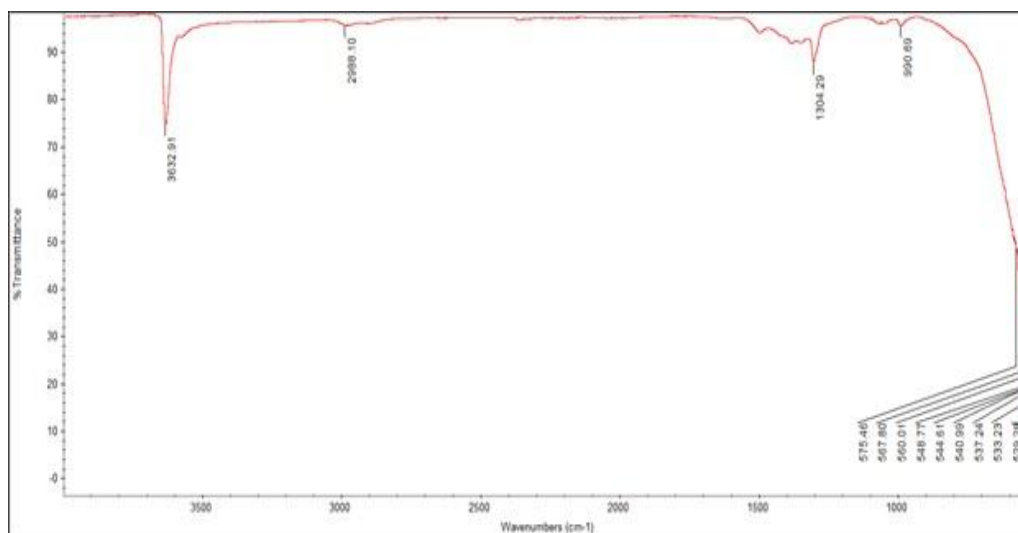


Fig 3.1a. FT – IR spectrum of nano nickel oxide

Fig 3.1b. revealed the spectra of poly vinyl pyrrolidone – Nickel oxide nanocomposite where the large broad bands appeared at 3638 cm⁻¹ which is due to the O-H stretch because of absorption of water molecule the peaks at 2908 cm⁻¹ C-H stretch, the peaks at 2358cm⁻¹ & 2330 cm⁻¹ is due to the (N-H) unsaturated amine, 1405 cm⁻¹ is due to C=O aromatic stretch , 1065 cm⁻¹ is due to C-O stretch, 1985 cm⁻¹ is due to N-H bending . C-H bending peaks peaks appeared from 891 to 608 cm⁻¹ and the peaks from 582 to 537 cm⁻¹ is due to the metallic stretch.

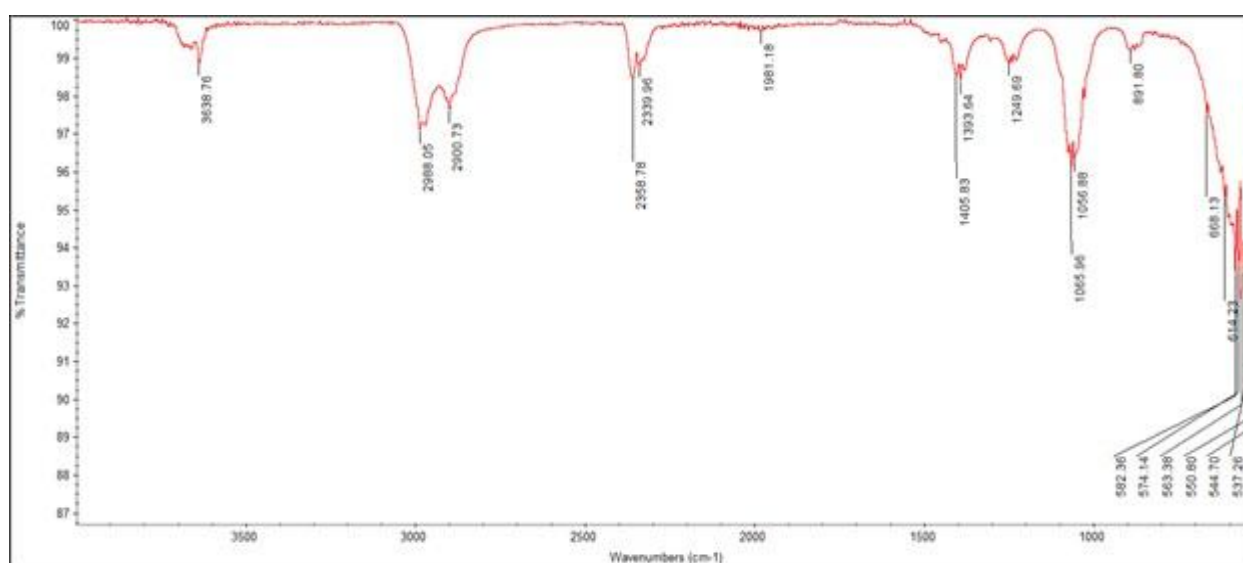


Fig 3.1b FT-IR spectrum of PVP- nanoNickel oxide

UV- vis absorption studies

UV-vis spectrum indicates blue shift in the bands which may be due to the presence of small sized nanoparticles.

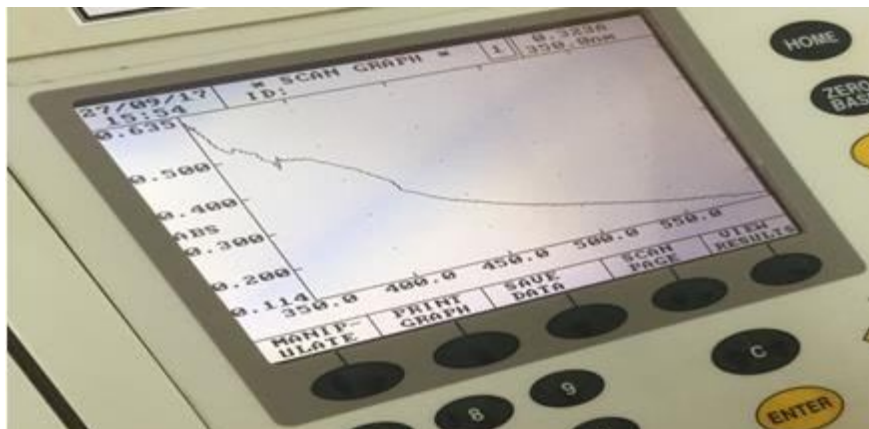


Fig 3.2 a . UV-visible spectrum of nanoNickel oxide

UV-vis absorption spectra of PVP- nanoNiO composites are shown in fig 3.2 b. The Fig 3.2 b shows that the broad and less symmetric absorption peaks were observed at 400 nm, due to blue shifted as compared to the bulk material. The blue shifting of effects are caused due to the quantum size effect of the broadening and assymetricity are due to the huge size distribution of synthesized materials and it was also confirmed to our SEM results.

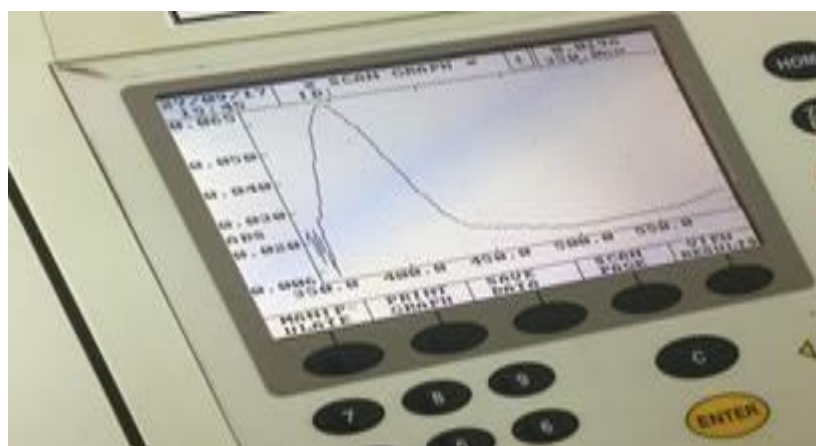


Fig 3.2 b. UV-visible spectrum of PVP- nanoNickel oxide composite.

SEM studies

SEM image clearly indicated that nano rod shape of nanoNiO particle were formed and they were agglomerated to form large size structures. Hence, the exact size and shape of the NiO nano particles were calculated by XRD. Fig.3.3b SEM image of PVP- nanoNickel oxide composites showed that cluster like structure and

the presence of such sharp crystal of Nickel oxide nanoparticles was incorporated with polymers like poly vinylpyrrolidone.

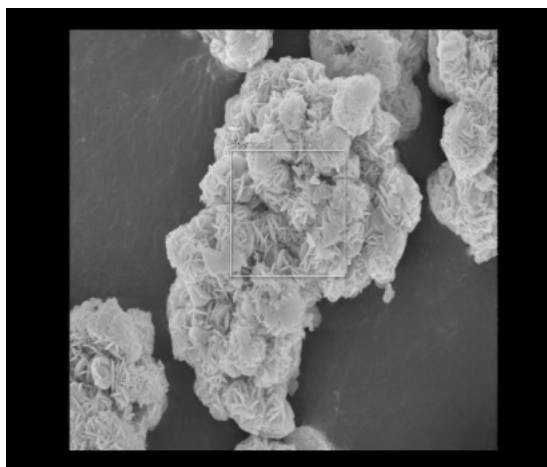


Fig3.3a SEM image of nanoNickel oxide

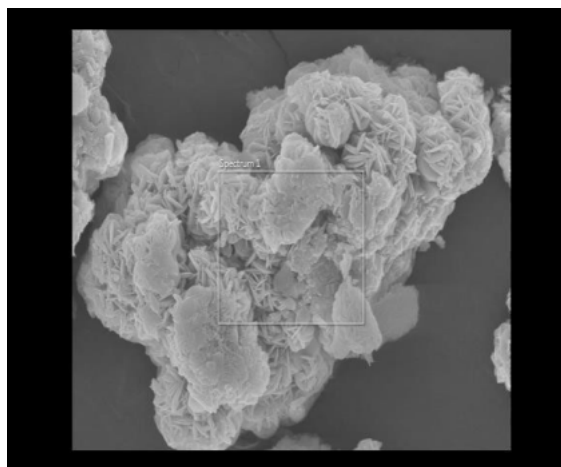


Fig 3.3b SEM image of PVP- Nickel oxide nanocomposite

Electrical properties of nanoNickel oxide and PVP-NiO nanocomposite.

Table 3.1 Electrical property of nanoNiO

Temperature °C	Electrical conductivity of nano NiO in $\mu\text{S}/\text{cm}$
25	54.5
35	68.4
45	86.8
55	114.4
65	158.0
75	221.8
85	264.5

Generally electrical conductivity might be increased or decreased depending upon nanofiller loading level. The electrical conductivity of nanoNickel oxide particle was measured at different temperature interval presented in Table 1. Electrical conductivity was increased with increased in temperature for nanoNickel oxide particles.

Table 3.2 Electrical property of PVP-NiO nanocomposite

Temperature in °C	Electrical conductivity of PVP- NiO nanocomposite in $\mu\text{S}/\text{cm}$
25	29.46
35	39.1
45	55.7
55	73.4
65	110.6
75	160.4
85	209.2

Electrical conductivity of PVP-nano NiO composite were lesser than the pure nano NiO in Table 3.2. The electrical conductivity of the nanocomposite was measured at different temperature interval and the results are presented in Table 3.2. Electrical conductivity of PVP- NiO nanocomposite was increased with the increase in temperature. Decrease in electrical conductivity of polymeric nanocomposite was expected due to the nanofiller aggregation. Therefore the polymer loading was adjusted between low to high wt% and it should be optimized for further studies.

4. CONCLUSION

PVP-Nickel oxide nanocomposites were successfully synthesized by in-situ polymerization method. Formation of polymer matrix increased the stability and homogeneity of the nanofiller in the nanocomposite. The results of FTIR spectra and SEM confirmed the formation of the composite and indicate an interaction between PVP and nickel oxide nanoparticles. The maximum sensitivity is observed for electrical conductivity of nanoNickel oxide particles. PVP-NiO nanocomposite was comparatively lesser electrical conductivity nature than nanoNickel oxide. From the above results we concluded that PVP- nanoNickel oxide composite also used for sensor applications. The as synthesized PVP-NiO nanocomposite showed good result in optical and electrical properties and hence this composite is a promising material for coating technology and semiconductor devices.

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