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.Synthesis and Morphological Study of Nickel Oxide Nanoparticle

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Abstract: *Nickel oxide (NiO) nanoparticles have been successfully synthesized by sol-gel method using ascorbic acid was used as a reducing reagent and ethylene glycol as a sol stabilizer and also served as a diffusion barrier. The characterization has been done with XRD, TEM, FTIR, and UV-Vis Spectroscopy. The particle size was determined from X-ray diffraction which was also confirmed by TEM. The band gap energy was calculated by UV-Vis-NIR and structural property by FT-IR spectroscopy. The results obtained from the study confirm the formation of nickel oxide nanomaterial with the particle size of 25 nm.*

Keywords: NiO, Sol-Gel Method, Band Gap

I. INTRODUCTION

From the past few years, researchers are attracted to the development of nanomaterial due to their excellent chemical, electrical and optical properties [1,2]. Hence, nanomaterials are widely used in photocatalysis [3], removal of toxic metals [4], photoelectrochemical cells [5], dye-sensitized cells [6], gas sensors [7], and electrochemical sensors [8]. One of the important factors of a nanoparticle is their synthesis with easy method, green chemistry, using cheap chemicals, and so on. This preparation can be done with different synthetic methods like sol-gel methods, hydrothermal, microemulsion, precipitation, microwave, etc. which led to the preparation of special engineering material [9,10]. Nowadays, many efforts have been concentrated on the synthesis of metal oxide nanomaterial due to its outstanding properties as comparedto bulk materials. The properties of nanomaterial are different from bulk one is due to its nature of atomic structure in the interfacial regions of the nanoparticle.

Nickel oxide is a NaCl-type antiferromagnetic oxide semiconductor with p-type conductivity films due to its wide band-gap energy range from 3.6 to 4.0 eV [11]. It is difficult to prepare size-homogeneous and well-dispersed NiO nanoparticles with lesser particles size. The commonly used nickel salts are nickel acetate, nickel chloride, nickel sulphate, nickel nitrate, and nickel citrate. Estelle et.al [12] studied the effect of these salts on the properties of NiO nanoparticles and reveals that nickel acetate precursor forms reduced NiO having high surface area and sponge morphology. Wang et al [13] prepared NiO nanocrystalline with an average particlediameter of 18–55 nm using a surfactant-mediated reagent.

In this paper, we disclose a simple method for the synthesis ofwell dispersed, sphere-shaped, highly stable nickel oxide nanoparticles by sol-gel method and found that the use of alkaline ascorbic acid in glycol atmosphere successfully formed the nickel oxide nanoparticles.

II. EXPERIMENTAL

2.1 Materials and Methods

All chemicals used were of analytical reagent grade purchased from Merck. Doubly distilled water was used throughout Fourier- Transform Infrared (FT-IR) were recorded at a range of 4000-400 cm^{-1} using a Shimadzu FTIR Spectrometer. The crystal structure was studied by a Brucker D8 advance X-ray Diffracto meter at UGC-DAE

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Consortium, Indore with Cu K radiation (λ = 1.54 Å) to check the phase system and purity of the sample.The diffuse reflectance of the nanocomposites was performed at UGC-DAE Consortium, Indore using the Perkin-Elmer USA model Lambda 950.The TEM used in this work is at SAIF, Punjab University Chandigarh.

2.2 Synthesis of Nickel Oxide Nanoparticles

20 ml of ethylene glycol and 0.1M of nickel acetate solution were stirred for 15 minutes. To that solution, alkaline ascorbic acid was added drop wise for a time period of 30 min. The molar ratio of NaOH to ascorbic acid was set to be 1:0.1 mole. After addition of the alkaline ascorbic acid to a metal salt solution, a green colour ppt. of nickel hydroxide was formed. The ppt. was then washed with double distilled water and then with alcohol. The ppt. was then dried in at oven at 250°C for 5 hours. The chemical reaction between nickel acetate and NaOH is as follows:

$$Ni(CH_3COO)_2 + 2\ NaOH \rightarrow Ni(OH)_2 + CH_3COONa$$

Nickel hydroxide decomposes by heating to nickel oxide as:

$$Ni(OH)_2 \rightarrow NiO + H_2O$$

III. RESULTS

3.1 XRD

The XRD pattern of NiO nanoparticles is as shown in fig. 3.1.The characteristic peaks of NiO nanoparticles occur at 2θvalues of 17.15º, 29.91º, 33.89º, 37.71º, and 44.72º corresponding to the miller indices of (111),(200) (220) (311)and (400), respectively [14]. All these diffraction peaks correspond to the face-centered cubic (FCC) crystalline structure of NiO which is according to the standard spectrum (JCPDS, No. 40-1191).The crystallite size of the nickel oxide nanoparticles was calculated by using the Scherrer equation and obtained to be 25 nm.

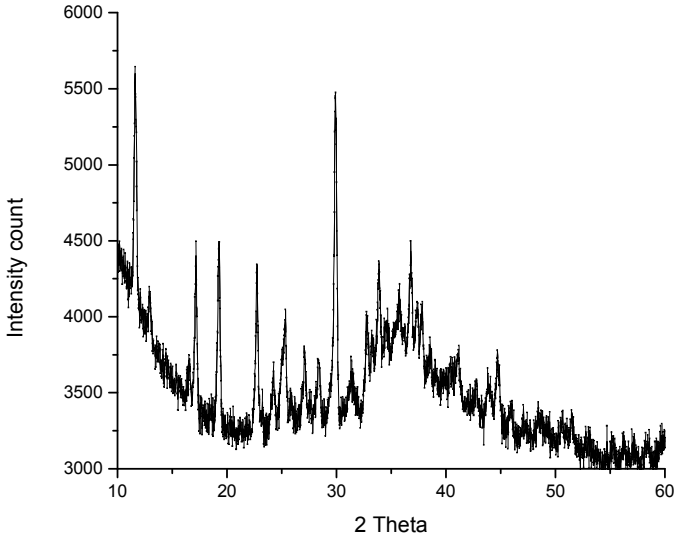

Figure 3.1: XRD pattern for NiO nanoparticle

3.2 TEM

Fig. 3.2 shows the TEM image of the NiO nanoparticles. It is seen from the image that the uniform NiO nanoparticles had narrow size distribution and rod shape with agglomeration. Most of the particle size shown to be in the range of 17-25 nm, which confirms from the XRD results. The specific surface area for NiO nano particles was calculated to be32.68 m^2/g. Some particles were shown to be a spherical shape with weak agglomeration. The agglomeration can be found because of the Vander Waals forces of attraction and magnetic interaction of the nanoparticles.

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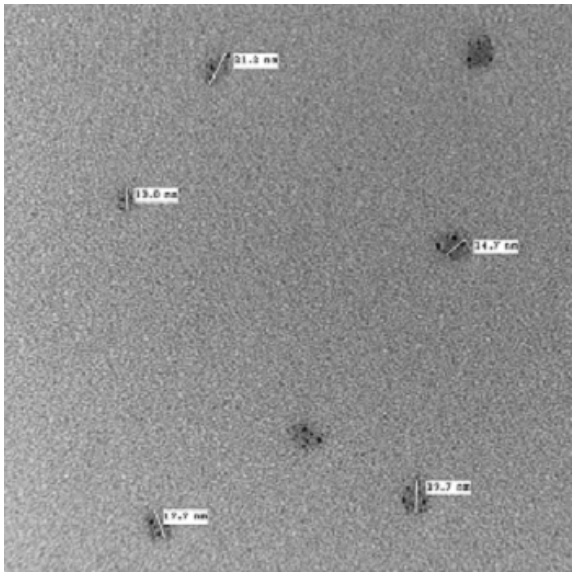

Figure 3.2a: TEM image for the NiO nanoparticle at 20nm

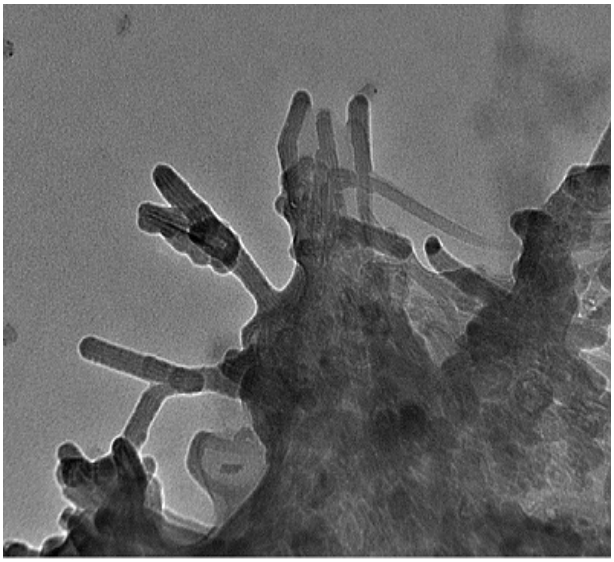

Figure 3.2b: TEM image for the NiO nanoparticle at 100 nm

3.3 FTIR

The IR spectrum of the precursor nickel oxide nanoparticle is shown in Fig. 3.3. The broad absorption band centered at 3639 cm^{-1}which is attributed due to the stretching vibrations $\upsilon_{(O-H)}$, and the band at 1604 cm^{-1} is attributed due to bending mode $\delta_{(H-O-H)}$. The band at 1327 cm^{-1} is primarily due to the bending vibration of ionic CO_3^{2-}. The three bands appearing around 1155 cm^{-1}, 952 cm^{-1}, 750 cm^{-1} confirm the presence of $\upsilon_{(C-O)}$ in the precursor. The strong band at 478 cm^{-1} corresponds to the bending vibration of NiO [14-16].

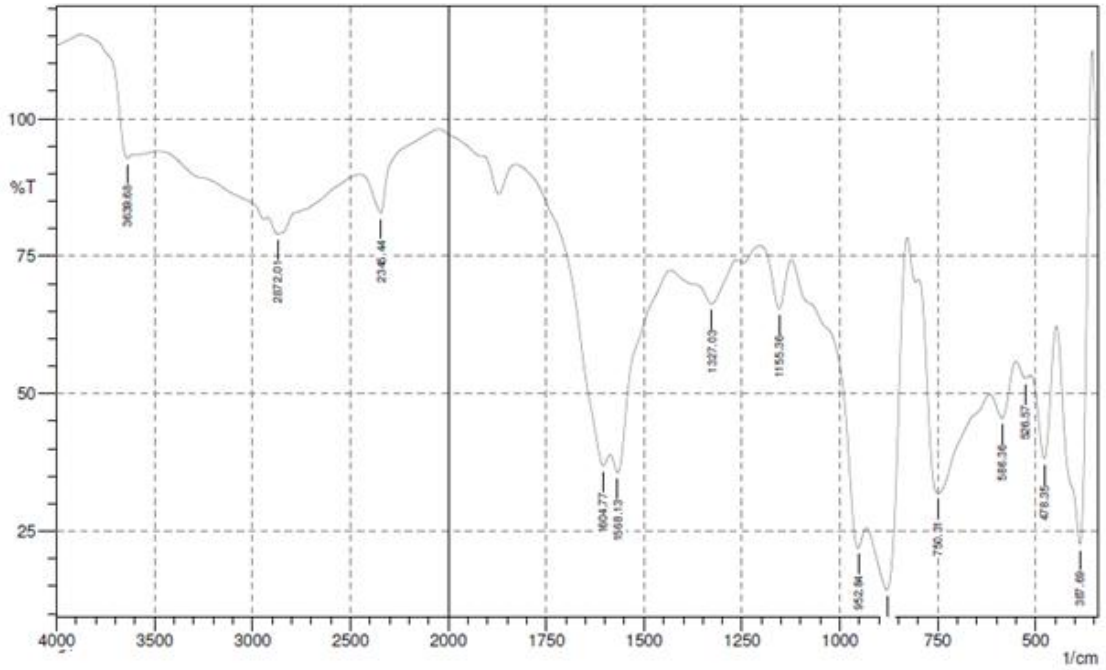

Figure 3.3: FTIR spectra for NiO nanoparticles

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3.4 Diffused Reflectance Spectra

Fig. 3.4a and b show the absorption spectra and Diffused Reflectance spectrum of nickel oxide nanoparticles sample. The absorption edges are seen to be shifted slightly towards the lower wave number (blue shift). This shift indicates an increase in the bandgap, which can be attributed to a decrease in particle size. The value of the absorption edge of NiO nanoparticle was 320 nm, the blue shift was observed due to the quantum confinement effects [14]. According to the data of the absorption spectra, the optical band gaps (Eg) of NiO nanoparticles can be estimated by using the following equation:

$$(Ahv)^n = B\left(hv - E_g\right) \ldots\ldots\ldots (3.8)$$

where hυ, A, B, and n are photo energy, absorbance, constant relative to the material and n = ½ or 2 for indirect transition and direct transition, respectively.

Hence, the optical bandgap for the absorption peak can be obtained by extrapolating the linear portion of the $(Ahv)^n - hv$ curve to zero shown in Fig. 3.4b. The bandgap is determined by extrapolating the linear portion of the plot to the energy axis. The corresponding bandgap energy calculated to be 3.87 eV [14] for sample, which was in good agreement with the value of 3.6- 4.0 eV for the NiO nanoparticles [17].

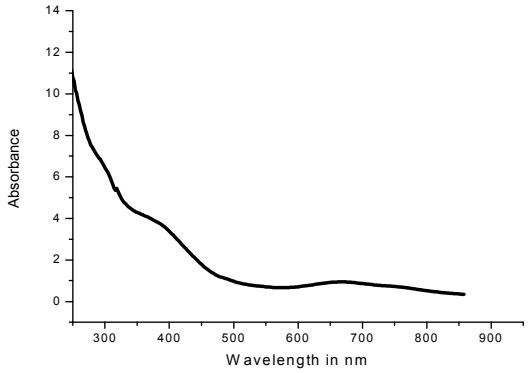

Fig. 3.4a UV-Vis absorption spectrum of NiO nanoparticles

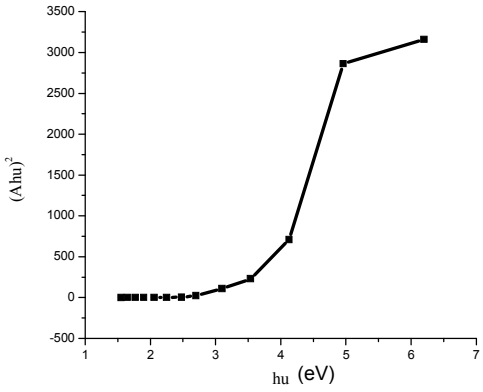

Fig. 3.4b $(Ahυ)^n$ versus (hυ) spectra of as-synthesized NiO nanoparticles.

IV. Conclusion

NiO nanoparticles were triumphingly synthesized by sol-gel method with particle size 25 nm which was confirmed by TEM study. The spectroscopic analysis of NiO nanoparticle has been characterized by UV-Vis-NIR and FTIR techniques. The band gap energy was found to be calculated as 3.87 eV.

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