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Synthesis and Characterization of Nickel Oxide based Nanocomposite Material

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ABSTRACT
Nickel oxide (NiO) nanoparticles and its composite with Polyaniline have been synthesized via sol-gel method. In this method, ascorbic acid was used as a reducing reagent and ethylene glycol was used as a sol stabilizer and also served as a diffusion barrier. The synthesized precursors were heated at 250oC for 5 h to obtain the nickel oxide nanoparticles. The synthesized sample were investigated by UV-Vis and FT-IR spectroscopy. The results obtained confirm the presence of nickel oxide nanopowders and its composites produced during sol-gel method.
Keywords - NiO-PANInanocomposites, polyaniline

I. INTRODUCTION

Nanomaterials exhibit significantly mechanical, electronic, magnetic, thermal, catalytic properties, and optical properties in comparison with their bulk counterparts, and have extensively attracted interests [1,2]. NiO nanoparticles may have many applications such as in the manufacture of electrochromic, films, magnetic materials, p-type transparent conducting films, gas sensors, catalyst, alkaline batteries cathode, and solid oxide fuel cells anode [1–10].Typically,preparation of nickel involves the solution phase chemistry route, which in theory should provide multiple, simple ways to control the morphology, particle size and desirable crystalline phase. Ideally, the process should be amenable to scaling up. Nickel nanoparticles have been synthesized by reduction of metal salts using reducing agents such as $NaBH_4$[11-12], hydrazine [13–16] and polyols [17–21]. While these processes can produce spherical, stable nanoparticles without agglomeration; the synthesized particle surfaces are often found to be rough and exhibit spiky surface morphology [21–24].The uses and performance for a given property and application are, however, stronglyinfluenced by the crystalline structure, the morphology, and the size of the particles.

Therefore, it is very important to develop methods for the synthesis of nickel oxide nanoparticles in which the particle size and the crystal structure of the products can be controlled.The main focus of interest in the field of nanocomposite material is the production of high yield, low cost and high resistance to abrasion.The efficient polymerization of aniline is achieved only in an acidic medium, where aniline exists as an anilinium cation. A variety of inorganic and organic acids of different concentration have been used in the syntheses of PANI; the resulting PANI, protonated with various acids, differs in solubility, conductivity, and stability. For the present study, we have selected sulphuric acid in specific proportion to aniline, i.e. aniline sulphate was used as a monomer. The handling of solid aniline salt is preferred to liquid aniline from the point of view of toxic hazards. Peroxydisulfate is the most commonly used oxidant, and its ammonium salt was preferred to the potassium counterpart because of its better solubility in water. The polymerization is completed within 10 min at room temperature and within 1 h at 0–2 °C . The oxidation of aniline is exothermic so the temperature of the reaction mixturecan be used to monitor the progress of reaction [12–14].We disclose herein a method for the synthesis ofwell dispersed, sphere-shaped, highly stable nickel oxide nanoparticles by sol-gel method. We discover that the use of alkaline ascorbic acid in glycol atmosphere leads to the formation of nickel oxide nanoparticles.

II. EXPERIMETAL

2.1. CHEMICAL AND APPARATUS:

All chemicals used were of analytical reagent grade purchased from Merck. Doubly distilled water was used throughout.

2.2 Instruments:

Fourier- Transform infrared (FT-IR) were recorded at a range of 4000-400 cm^{-1} using a

Shimazdu FTIR Spectrometer. For UV-Visible absorption Spectra, the sample was dissolved in water/DMF and spectra were recorded on UV-Visible Shimadzu UV Spectrometer.

2.3 Synthesis of Nickel Oxide nanoparticles:

20 ml of ethylene glycol and 0.1M of nickel acetate solution was stirred for 15 minutes. To that solution alkaline ascorbic acid was added dropwise within a period of 30 min. the molar ratio of NaOH to ascorbic acid was set to be as 1:0.1 mole. After addition of alkaline ascorbic acid to metal salt solution, 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 at oven at 250oC 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:

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

2.4 Synthesis of Ni/PANI composite material:

Nanocomposite material of NiO have been synthesized by the sol-gel method. In 500ml of conical flask, 0.1M of nickel acetate and ethylene glycol was stirred for half an hour. To this viscous solution, 1M of NaOH added drop wise. The broad ppt. of $Ni(OH)_2$ forms in the solution. The solution was stirred for 1 hour and then in another flask 10ml of aniline was uniformly stirred with 1M H_2SO_4 and the synthesized $Ni(OH)_2$ was then poured to the aniline solution. Polymerization was carried out in ice-cold condition in presence of potassium peroxydisulfate. The green colour ppt. forms on addition of oxidizing agent. The solution was stirred 5 hours and then washed with distilled water 3 times to remove impurity if present and then the ppt. was then filtered and dried in furnace.

III. RESULT:

UV-Visible Spectroscopy:

The absorbance spectra of the synthesized metal oxide Polyaniline was recorded using the double beam spectrophotometer of Shimazdu UV-Visible spectrophotometer. The spectra shows two characteristics peaks at a wavelength of 370nm and 630nm. The first peak corresponds to the formation of nickel oxide while the latter on peaks at 630nm corresponds to the formation of Polyaniline.. It can be seen from the figure that for different temperature maximum absorbance occurs at 640nm due to the transition of two benzenoid rings to the Quinoid rings of the PANI chain Apart from that, the observed spectra was quite differ from the standard value, this reveals that there occur a shift of the polymeric material. . The sample exhibit absorption spectra near 370nm shows the π- conjuagated system transition of the oxide nanoparticles

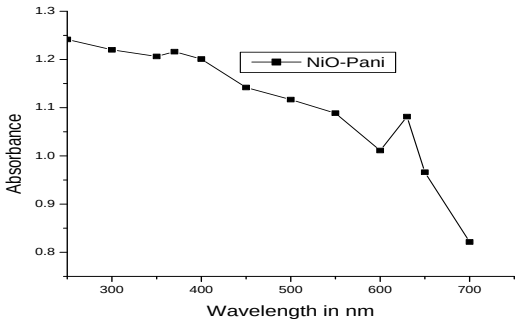

Fig1. Absorbance spectra of NiO-PANI nanocomposite material

FTIR Spectroscopy:

The IR spectrum of the precursor nickel oxide nanoparticle is shown in Fig 2(a). The broad absorption band centered at 3439 cm^{-1} is attributed to the band O–H stretching vibrations, and the band at 1604 cm^{-1} is attributed to bending mode (H–O–H). The band at 1327 cm^{-1} is primarily dueto the banding vibration of ionic CO_3^{2-}. The three bands appearing around 1162, 952, 750 cm^{-1} confirm the presence of C–O in the precursor. The strong band at 478 cm^{-1}corresponds to thebanding vibration of NiO.Fig 2b. exhibits the IR spectrum of its composite material. The characteristic peaks of NiO/PANi nanocomposite occur at 3637, 2953, 1573, 1230, 1159,948, 748, 588 and 480 cm^{-1}. The strong absorption peak at 3637 and 2235 cm^{-1} are assigned to the N-H stretching vibration of amino group of PolyanilineThe peaks at 1573 $cm^{-1}$are attributed to the characteristic C=C stretching of theQuinoid rings of polyaniline; the peaks at 1241 cm^{-1}correspond to asymmetric C–N stretching modes of the benzenoid ring.The peak around 1159 $cm^{-1}$is associated with vibrational modes of N=Q=N (Q refers to the quinonic type rings), indicating that PANi is formed in our material.However, the characteristic peaks of NiO can be observed at higher wavenumbers 480 cm^{-1} indicating that there is an interaction between NiO nanoparticles and PANi chain.

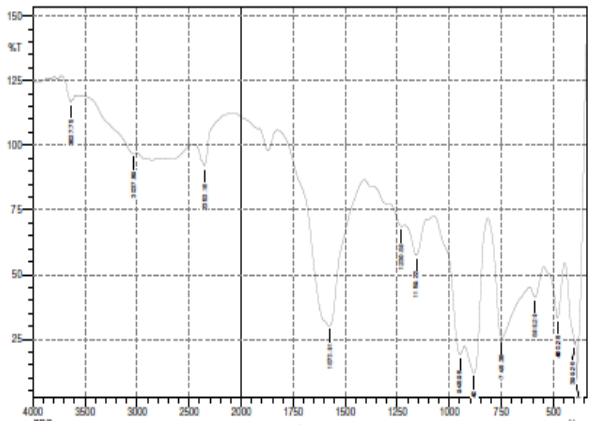

Fig 2 a. FTIR spectra for NiO nanoparticles

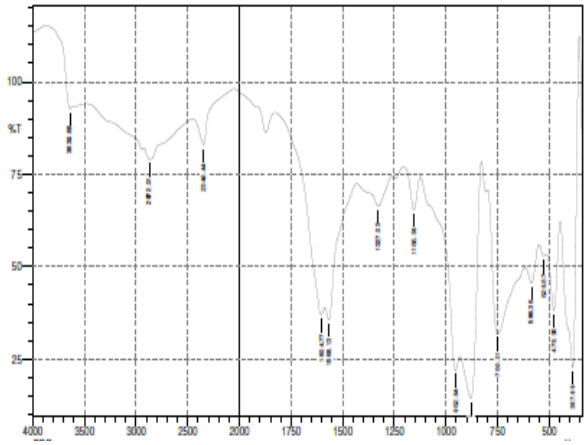

Fig 2b. FTIR spectra for NiO/PANI composite material

III. CONCLUSION:

We have synthesized Polyaniline nanocomposite material by sol-gel method. Polymerization carried out of aniline in the presence of potassium peroxydisulfate in acidic medium which was then bind up with nickel salt in alkali medium. The spectroscopic analysis of PANI-NiO nanocomposites have been characterized by UV-Visible and FTIR techniques. The UV-Vis results demonstrates the formation of Polyaniline in the presence of NiO nanoparticles.

IV. ACKNOWLEDGEMENT:

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