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Synthesis of NiO nanoparticles by sol-gel based catalyzed method

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Abstract

During the last few years, a vast number of research works have been carried out in the field of nanotechnology. Amongst all the works under investigation, NiO nanoparticles are largely studied owing due to its low-cost, ease of availability of raw materials, good electrical and magnetic properties as well as being used in a wide variety of applications. In this paper, NiO nanoparticles have been synthesized by sol-gel method under based catalyzed system using nickel nitrate hexa hydrate and potassium hydroxide as the precursors. The size and shape of the synthesized NiO nanoparticles were characterized by scanning electron microscopy (SEM). From SEM studies, it was found that NiO nanoparticles were ~71-115 nm in size and the crystallite size strongly depends upon the calcination time. The structural properties of NiO nanoparticles were studied by X-Ray diffraction (XRD) method. XRD studies showed the formation of well crystallized NiO phase having good purity. AC-Impedance study were carried out to determine their resistance and conductance properties. The synthesized NiO was found to have very good values which revealed that it is suitable to be used to create and fabricate a number of materials in the industry.

Keywords

NiO, nanoparticles, SEM, calcination, XRD, AC-Impedance

Introduction

Nanophase engineering has grown exponentially over the last decade. The discovery of nano metal oxides has created a huge fuzz with respect to their advanced mechanical, catalytic, as well

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as electronic properties in small materials **[1-5]**. Thus the quest to infuse these properties to develop small devices has led many researchers to create an effective bridge between bulk materials and atomic sized materials as well as nanomaterials. A large number of methods have been devised to synthesize and design new nanoparticles. Some of the techniques involved in developing new nano sized materials are chemical precipitation, sol-gel, electro-spinning, ultrasonic-spray pyrolysis, electrodeposition, chemical vapour deposition, hydrothermal synthesis, solid state method and so on **[6-11]**. Amongst all the metal oxides investigated, NiO nanoparticles are widely studied due to their interesting properties such as its large surface area, good optical, thermal, electrical as well as magnetic properties **[12,13]**. NiO nano particles are known to exhibit various types of magnetic behavior such as paramagnetism, ferromagnetism, anti- ferromagnetism based upon the size and shape of the nanoparticles formed as well as the procedure involved in the synthesis of the nanoparticles **[11]**. Besides this, NiO nano particles is an important class of the transition metal oxides as it has a band gap of 4 eV which makes it vitally suitable for use in p and n type semiconductors and for other applications in the organic electronic industry **[14]**. The discovery of nanomaterials especially NiO nanoparticles have thus paved a way in the field of medicine, biochemistry, colloid chemistry, p-type transparent conducting films, gas sensors, catalyst, alkaline batteries cathode aerosol research, photo-voltaics, super-capacitors and like-wise in various array of applications **[15-18]**.

The main aim of this paper, is to synthesize NiO nanoparticles by sol-gel based catalyzed method using KOH which is not only easily available but also cost effective.

Experiment

Chemicals used

Nickel nitrate hexa hydrate ($Ni(NO_3)_2.6H_2O$), KOH. All the chemicals used were of analytical grade and used as such without any further purification. Deionized water was used in this synthesis.

Instruments used

SEM images for the synthesized sample by sol gel based catalyzed method was carried out on a JEOL JSM 6390 Scanning Electron Microscope. While, XRD study was performed on a Shimadzu XRD 6000 X-Ray diffractometer, whereas, AC-Impedance study was recorded on a

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CH660 D Electrochemical work station. Here in AC-Impedance study, NiO nanopowder used was made as a paste by mixing it with polyvinyl alcohol **(PVA)** and ethanol. The paste was then coated onto the working electrode and was dipped in a solution containing 0.01 M dil H_2SO_4. The solution consisted of two electrodes *viz* platinum electrode and saturated calomel electrode.

General Procedure

The nickel salts were dissolved in deionized water to form a solution of 0.3M concentration. 2M KOH was then added drop-wise into the solution and stirred continuously on a magnetic stirrer at room temperature (RT). Subsequently, the green precipitate formed was filtered out and rinsed with water. The filtered compound was then dried at ~80ºC overnight. $Ni(OH)_2$ is then converted to NiO by calcinating the sample at 350 ºC. The resultant product obtained was greyish black in colour. Nickel hydroxide gel was formed by the following reaction step.

$$Ni(NO_3)_2 + 2KOH \rightarrow Ni(OH)_2 + 2KNO_3$$

Results

SEM studies

Fig 1 shows the SEM image of the compound synthesized by base catalyzed sol-gel method. It was observed that most of the particle sizes were in the range of 70-115 nm respectively. It can be noted that potassium has higher reactivity to form ion and thus reacts with the nickel precursors to form $Ni(OH)_2$.

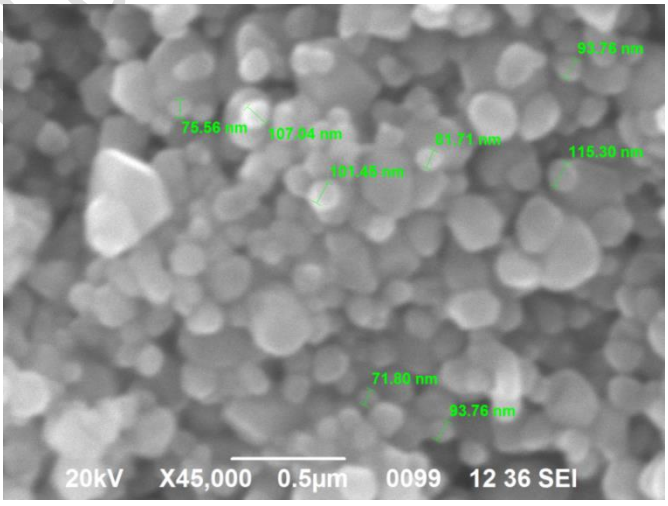

Fig1: SEM image of NiO nanoparticles synthesized by sol-gel based catalyzed method

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XRD studies

The XRD pattern of the synthesized NiO nanoparticles calcined at 350º C is shown in **Fig 2.** From the XRD pattern, it can be noted that the synthesized compound is crystalline in nature. The annealing temperature (350ºC) show the enhanced crystalline structure of NiO which can be seen due to presence of well defined peaks. When the synthesized compound was calcined to 350ºC, the crystal phase of NiO was observed from the height of the main peaks at angles 2θ about 37º, 43º and 62.5º respectively which is found to be in good agreement as compared with the standard XRD table (JCPDS Card No. 22-1189). The average crystallite sizes (D) of the synthesized NiO nanoparticles were calculated using the Debye-Scherer equation **[19]** which is as follows

$$D = k\lambda/\beta\cos\theta \qquad \rightarrow \text{equation 1}$$

where D is the crystallite size, λ is the wavelength of X-ray used, β is the full width in radiation at half-maximum of the peak, and θ is the Bragg angle of X-ray diffraction peak, k is the Scherer constant of the order of 0.9 related to crystallite shape. Crystallite size of the nanoparticles confirms the formation of the nanocrystalline structure. The sizes of nanoparticles are found to be in the range between 70 nm and 115 nm. The grains are distributed uniformly over the entire surface and is in good agreement with the XRD results. The crystallite size are known to increase with an increase in the calcination temperature. Literarure reports states that in NiO nanoparticles there are a number of dangling bonds that are related to nickel-oxygen defects at the grain boundaries. These defects are responsible for the formation of larger NiO grains formed at higher temperature which further results in increased crystallite size **[20, 21]**. Furthermore, reports have also mentioned that high calcination temperature gives rise to high particle size thus increasing the band gap values **[10].** Anandan K and Rajendran V. in their work mentioned that as the annealing temperature is increased, the grain size and band gap values also increases. Thus, it can be clearly said that the band gap is dependent directly on the grain size of NiO nanoparticles which shows more or less linear dependence on the applied magnetic field. Therefore, NiO nanoparticles show paramagnetic behavior [22]. It can thus be said that the formation of Ni and NiO nanoparticles as well as their morphological features are largely dependent on the

calcination temperature *ie.* higher calcination temperatures, results in higher intensities of the diffraction peaks of NiO nanoparticles as well as better degree of crystallinity.

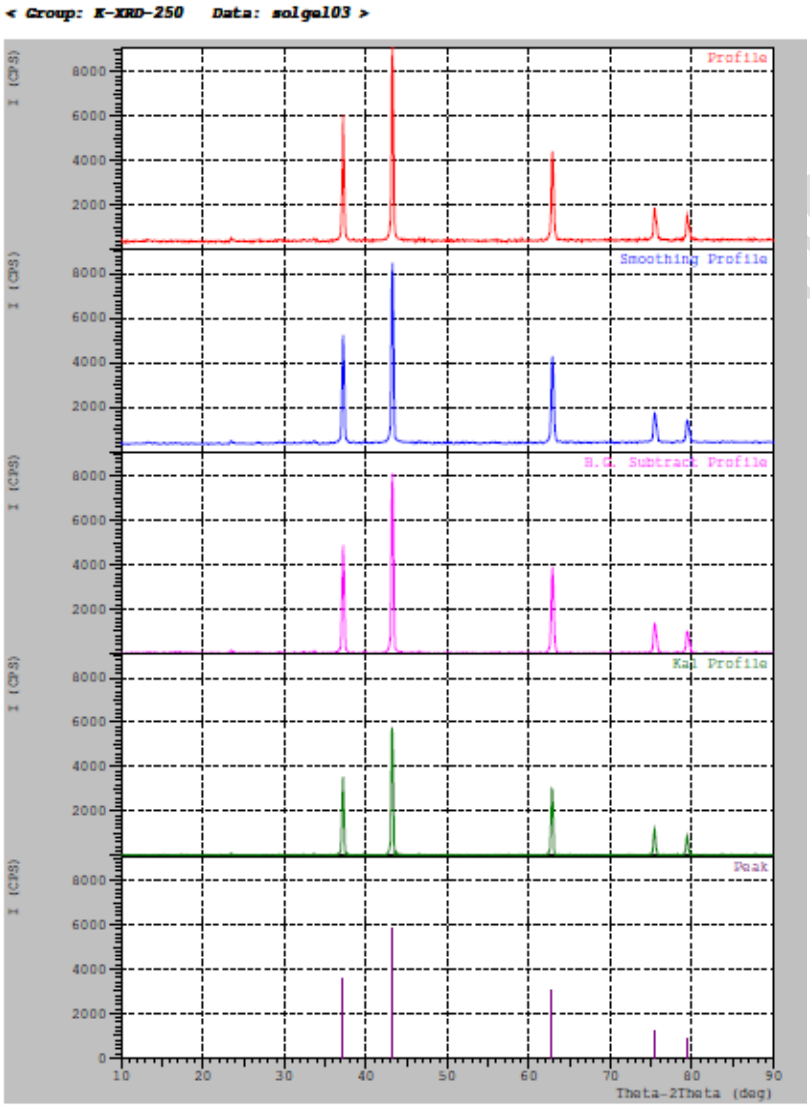

Fig 2: XRD image of NiO nanoparticles synthesized by sol-gel based catalyzed method

Particle size distribution

Particle sizes of the synthesized NiO nanoparticles were analyzed using particle size analyzer. A graph of the number of NiO nanoparticles *versus* the size of the particles is plotted as seen in **Fig 3**. It is observed that most of the particles are in the range of 71.8-115.3 nm respectively. All particles showed narrow size distribution and uniform shape.

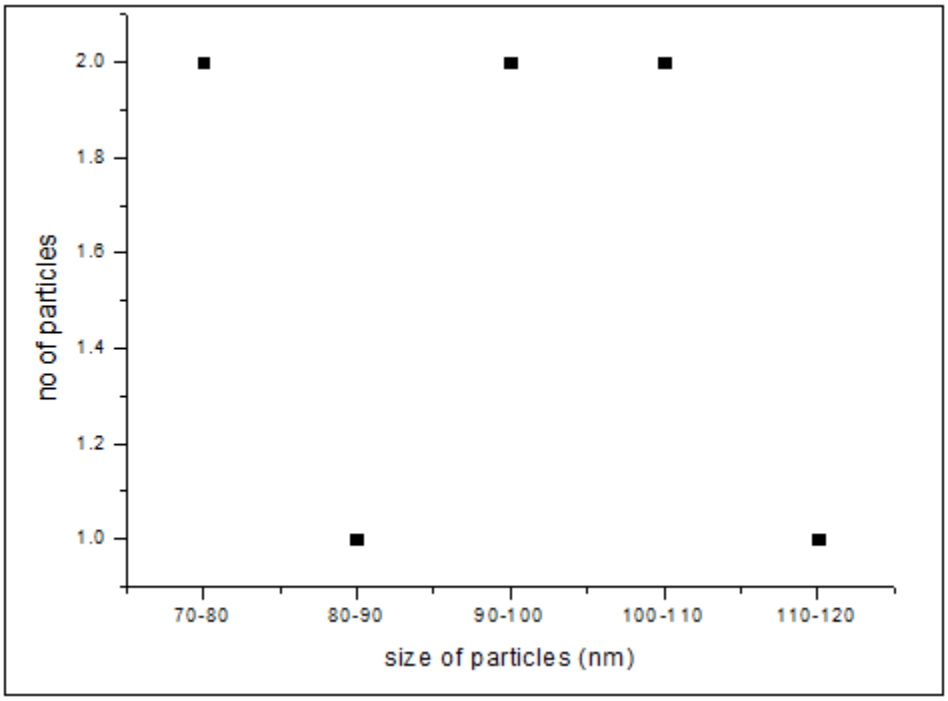

Fig 3: Distribution of NiO nanoparticles synthesized by sol-gel based catalyzed method

AC-Impedance studies

Surfactant	Resistance (Ω)	Conductance (mF)
Sol-gel process under base catalyzed method	452.6	9.679×10^{-3}

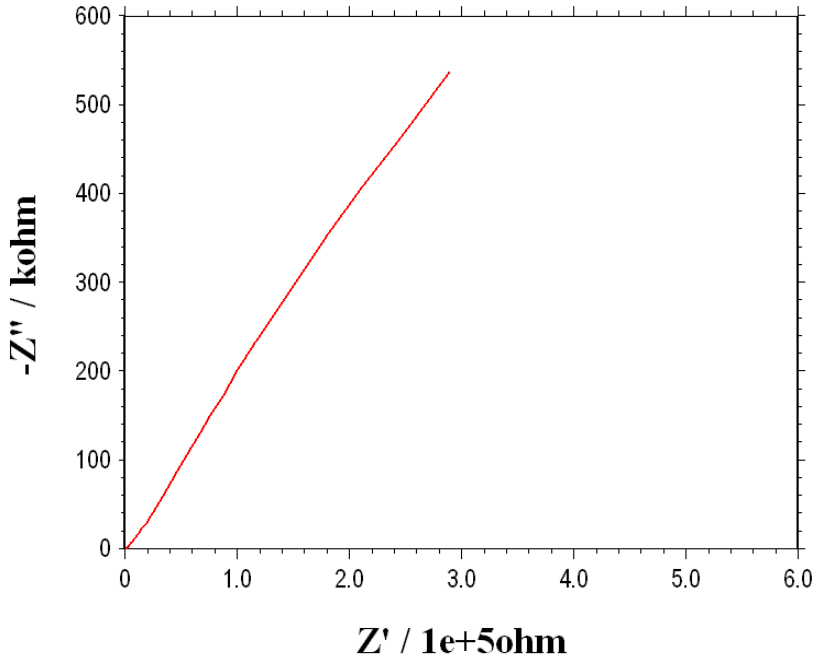

Fig 4: AC-Impedance study of NiO nanoparticles synthesized by sol-gel based catalyzed method

AC-Impedance study of the synthesized NiO nanoparticles showed very good resistance (452.6 Ω) as well as high conductance (9.679 $x10^{-3}$ mF) which confirms that it has excellent electrical resistivity and conductance properties thereby enhancing its potential use for applications in a wide range of fields.

Conclusion

From the above work, we can conclude that NiO nanoparticles were successfully synthesized by based catalyzed sol-gel method using nickel nitrate and KOH. NiO nanoparticles were annealed at 350ºC and were found to be ~71-115 nm in size as observed from the SEM. It can be concluded that higher calcination temperatures are responsible for the stability of NiO nanoparticles. The particle size distribution showed narrow size distribution and uniform shape. Whereas, AC-Impedance studies reported excellent resistance and conductance values which confirms their good electrical resistivity as well as conductivity properties. Overall it can be concluded that the synthesized NiO nanoparticles can be used for further electronic applications in various industries.

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