



Structural Characterization of NiO Nanoparticles and its Bio-applications

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Abstract:

Nanostructured nickel oxide with unique optical, electrical and molecular properties along with desired functionalities and surface charge properties provide interesting platforms for bioapplications. Nickel oxide nanoparticles were synthesized by a sol-gel process, and the reaction conditions were optimized to provide good biological properties and applications. The effect of various synthetic reaction and heat treatment conditions on the structural properties of NiO powder was studied. Physical characterizations of NiO are investigated by XRD and TEM. The NiO particle calcinated at 750°C for 2h has a higher surface area and uniform particle size distribution. XRD revealed the NiO nanoparticles have a crystalline bunsenite structure. The surface morphology of the NiO nanoparticles exhibited nanocrystalline grains with a randomly oriented morphology.

Key words: Nanoparticles, XRD, TEM analysis.

Introduction:

Nanostructured metal oxides (NMOs) have recently become important as materials that provide an effective surface for biomolecule immobilization with desired orientation, better conformation and high biological activity resulting in enhanced sensing characteristics. Nanometer-scale materials with the size of 1 to 100 nm have attracted considerable interest in recent years due to the departure of properties from bulk phases arising from quantum size effects. Living organisms are built of cells that are typically 10 μm across. However, the cell parts are much smaller and are in the sub-micron size domain. Even smaller are the proteins with a typical size of just 5 nm, which is comparable with the dimensions of smallest manmade nanoparticles. This simple size comparison gives an idea of using nanoparticles as very small probes that would allow us to spy at the cellular machinery without introducing too much interference. Understanding of biological processes on the nano scale level is a strong driving force behind development of nanotechnology. Nanoparticles exist in the same size domain as proteins makes nano materials suitable for bio tagging or labelling. In order to interact with biological target, a biological or molecular coating or layer acting as a bioinorganic interface should be attached to the nanoparticle. Examples of





biological coatings may include antibodies, biopolymers like collagen or monolayers of small molecules that make the nanoparticles biocompatible. In addition, as optical detection techniques are wide spread in biological research, nanoparticles should either fluoresce or change their optical properties.

Nickel oxide (NiO) is a technologically important material with applications in catalysts [1], electrochromic film [2–4], Biosensors[5,6], fuel cell [7], anode of organic light-emitting diodes [8], magnetic materials [9–11] and thermoelectric materials [12], owing to its p-type conductivity, wide band gap ranging from 3.6eV to 4.0 eV [13], excellent chemical stability, electrical and optical properties [14]. This transition metal oxide, when falling in the nanosized regime, is expected to lead to even more attractive biological applications. On the other hand, at room temperature, stoichiometric NiO is an insulator. Therefore, enhancing the electrical conductivity of NiO is an important issue for improving the applicability of NiO [15–17].

Several approaches have been used to prepare NiO nanoparticles such as pulsed laser deposition [18], sputtering [19], e-beam evaporation [20], electrochemical method [21] and sol-gel method [22–25]. Among these, the sol-gel method is the most cost-effective for producing large-area films, and provides excellent control of the composition and homogeneity. On the other hand, it is still challenging to develop simple, fast and versatile methods for the synthesis of highly conducting NiO nanoparticles.

Material and methods:

1. Experimental

Nickel carbonate (99%), sodium hydroxide (99%), starch (99%) were purchased from Merck. X-ray diffraction (XRD) analysis was conducted on Philips PW1710 automatic X-ray diffractometer with Cu-K α radiation ($\lambda=1.5404\text{\AA}$), with a scanning speed of 10°min^{-1} . TEM analysis of NiO was carried out on Phillips model-CM200 with resolution 2.4\AA .

1.1. Synthesis of NiO nanoparticles

Nickel oxide (NiO) nanoparticles were prepared by sol-gel process [26] in which, 0.1M nickel carbonate was added in 100ml starch solution and the mixture was stirred for half an hour. Then ammonia was added drop wise in the solution under constant stirring. After complete addition of ammonia, the solution was allowed to settle for overnight and then filtered using membrane filtration assembly, washed using deionized water and ethanol to remove the impurities and then dried at 80°C in hot air oven. Dried sample was treated at different temperatures in order to maintain the stability of compound. The colour of the sample was changed from green to faint gray at 100°C to 750°C .





Results and discussion:

1.2. XRD analysis

The nickel oxide nanoparticles were analyzed by X-ray diffraction technique to study structural identification and changes in the crystallinity. The XRD pattern of nickel oxide is shown in Fig. 1. It is found that NiO was polycrystalline consisting of NiO cubic phase, comprising a strong reflection along (1 1 1) plane and a weak reflection along (2 2 2) plane. The 'd' values of the XRD reflection were compared with the standard 'd' values. Good agreement between the observed and standard 'd' values confirms that the material is NiO (cubic). The magnitude of major XRD peak corresponding to (1 1 1) plane and value of area under the peak slightly ameliorates. The grain size of the NiO crystallites (mean crystallite diameter) was calculated for major reflex (1 1 1) using the well-known Scherrer's formula by assuming the factors, viz. instrumental broadening, distortion of lattice, etc. are common among all the samples: $D_{XRD} = 0.9\lambda / (\beta \cos \theta)$

Where D_{XRD} is the average crystalline size, λ is the wavelength of CuK α , β is the full width at half maximum of the diffraction peak, and θ is the Bragg's angle. The crystalline peaks at $2\theta = 37.53^\circ$, 43.60° , 63.09° , 75.67° , 79.58° which have been identified as peaks of single phase cubic structure of NiO with diffracting planes (111), (200), (220), (311), (222) respectively matched with JCPDS file 73-1523 [27-28].

1.3. TEM analysis

Morphology of NiO nanoparticles was studied by transmission electron microscope (TEM) as shown in figure 2B. Figure shows the particle morphology of NiO at high resolution. The particles are mostly irregular spherical shape with a nanosize range 20 nm. Some particles are found as agglomerated surface are observed. TEM image of NiO indicated that the prepared NiO nanoparticles have spherical morphology and average particle size of 20nm which is consistent with that estimated from the XRD data. From the TEM image of NiO, it could be concluded that this preparation method is appropriate to obtain the NiO nanoparticles with very small size [29-30]. Some distortion was observed due to self alignment orientation taking place due to the presence of weak interactions.

1.4. Biological applications of NiO:

An inspiration to science from the time of Faraday, today NiO nanoparticles are being used for an ever-growing number of applications. A field that has showed fast growth over the past decades is the use of nickel oxide nanoparticles in biology or life sciences. These bioapplications can be classified into four areas:

1] Cancer therapy:



Photodynamic cancer therapy is based on the destruction of the cancer cells by laser generated atomic oxygen, which is cytotoxic. A greater quantity of a special dye that is used to generate the atomic oxygen is taken in by the cancer cells when compared with a healthy tissue. Hence, only the cancer cells are destroyed then exposed to a laser radiation. Unfortunately, the remaining dye molecules migrate to the skin and the eyes and make the patient very sensitive to the daylight exposure. This effect can last for up to six weeks. To avoid this side effect, the hydrophobic version of the dye molecule was enclosed inside a porous NiO nanoparticles [31-32]. **2] Drug delivery:**

Secondly, NiO nanoparticles can serve as carriers for drug and gene delivery [33-34]. Biologically active molecules adsorbed on the particle surfaces can be guided inside cells and released. DNA delivery, for instance, is the basis for gene therapy.

3] Sensors:

NiO nanoparticles can also be used as sensors. Their optical properties can change upon binding to certain molecules, allowing the detection and quantification of analytes. The absorption spectra of NiO nanoparticles change drastically when several particles come close to each other. In the business of colloids aggregation is actually rather annoying but it can be exploited for very sensitive DNA detection, even of a single-base mismatch.

4] Labelling:

For labelling, certain properties of the particles are exploited to generate contrast. For example in transmission electron microscopy, the strong electron absorbing properties of NiO nanoparticles make them suitable as a stain for samples with poor contrast, such as tissue samples. Their small size and the possibility of functionalising the particles, for instance with antibodies (immunostaining), mean that they also provide extremely high spatial resolution and specificity in many labelling applications. Similarly, the particles' optical properties-strong absorption, scattering and especially plasmon resonance-make them of value for a large variety of light-based techniques including combined schemes such as photothermal or photo-acoustic imaging [35].

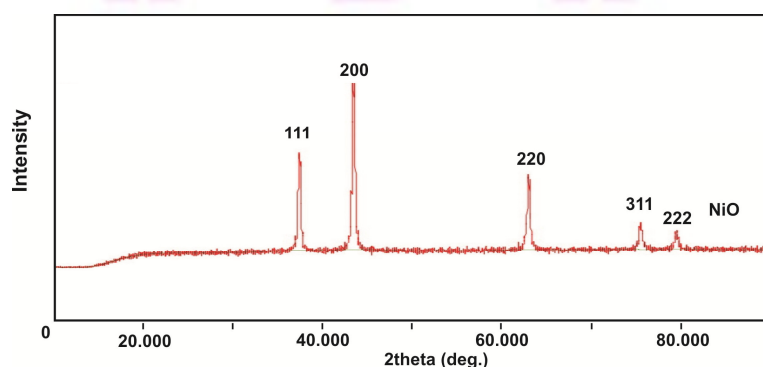


Figure.1- XRD of NiO

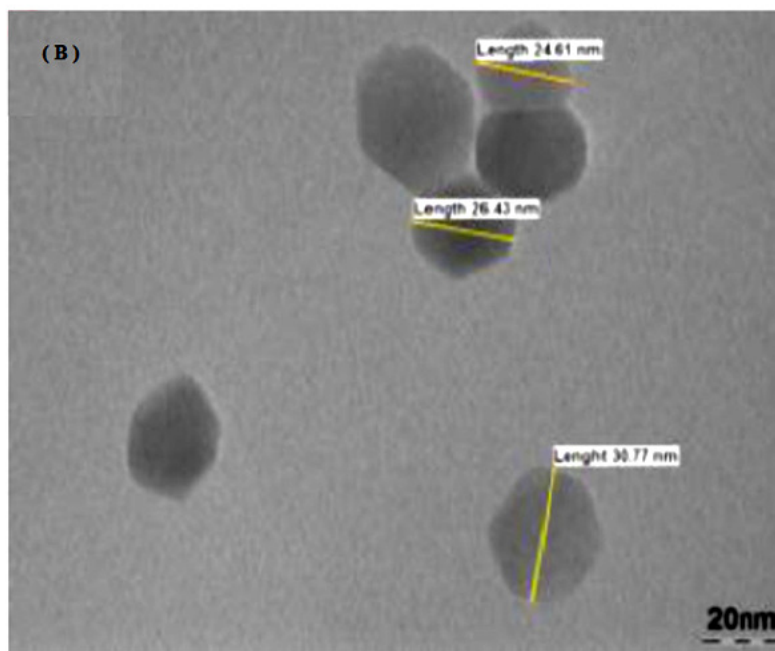


Figure.2- TEM of NiO

Conclusion:

XRD spectra of NiO shows cubic phase with orientation along (1 1 1) direction. TEM image of NiO indicated that the prepared NiO nanoparticles have spherical morphology and average particle size of 20nm.

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