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Characterization and DC Conductivity Studies of NiO Nano Particles

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ABSTRACT
In this paper nickel oxide, nanoparticles were synthesized by a low temperature solution combustion method due to simple, easy, and ecofriendly method. The synthesized nanoparticles were characterized by powder X-ray diffraction, (to confirm crystallinity) Fourier transform-infrared, (to identify the chemical bonds and to detect the functional groups) scanning election microscopy (to analyze the structure and morphology), and energy dispersive x-rays (for chemical analysis on nanometer scale) as a part of optical and morphological studies. Further, the dc conductivity of NiO nanoparticles was measured by two-probe technique in the temperature range 35-200°C.

Key words: *Nanoparticles, Solution combustion synthesis, X-ray diffraction, Field-emission scanning electron microscopy, Fourier transform-infrared, Energy dispersive X-ray, Debye sherror formula.*

1. INTRODUCTION

The technology that focuses characterization, application devices at nanoscale is known as nano on the synthesis technology. It has a wide range of disciplines in chemistry, materials, sensors, electronics, information storage, communication, protection measurements of the environment, aerospace, biological systems, drug delivery systems, etc. The window that opens the applications of nanotechnology is, of course, nanomaterials which have critical dimensions <100 mm they show very interesting and amazing properties a part from their bulk counterparts. By the process of synthesis, the atomic structure, size confinement, composition, microstructure, defects, and interfaces can be tailored on which the unique properties of nanomaterials depend. To achieve the desired coordination environment, size, shape of nanostructures, the synthesis process plays a very important role. In general, bottom-up technique and top-down technique are two main approaches for the production of nanomaterials where bottom-up approach continues from atomic or molecular scale to nanoscale, whereas the top-down approach leads to nanoscale from macro scale. Here, we have adopted liquid solution combustion synthesis to produce the nanoscale particles of nickel oxide.

Nowadays, studies are going on nanostructured metal oxides due to their wide application in different fields of science. Among various metal oxide, Nio is used for catalytic and magnetic, and conductivity applications. The present work deals with synthesis and characterization of nickel oxide nanomaterial and its conductivity variation with temperature. The chemical composition of nickel content is 78.55% and that of oxygen is 21.40%.The density and molar mass of NiO nanoparticles are 6.679 g cm^{-3} and 74.71 g $mole^{-1}$, respectively. The melting point is nearly 1955°C. In literature, it was found that NiO nanoparticles can be prepared by air calcinations of $Ni(OH)_2$ and by homogeneous precipitation method. In our study, NiO nanoparticles were synthesized by a low solution combustion method and characterized by X-ray diffraction (XRD), Fourier transform-infrared (FTIR), scanning election microscopy (SEM), and energy dispersive X-rays (EDXs) to know the structure and crystallite size, to identify the chemical bonds in a molecule and to know the morphology and to know the purity of the element by chemical analysis [1].

Optical conductivity of metal oxides can be experimentally obtained from reflectivity and absorption measurements. Reflectivity is completely sized dependent with respect to transport properties, oxide materials ionic/electronic conductivity is influenced by the nanostructure of the solid. According to Boltzmann statistics, the number of electronic charge carriers in a metal oxide is a function of the band gap energy.

Mechanical properties that can be studied for metal oxides are sinterability, ductility, and superplasticity.

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With respect to bulk counterparts, the works have shared significant improvement in sintering with up to 600°K lower temperatures.

In the process of chemical properties, metal oxides are used for both their redox and acid/base properties in the context of absorption and catalysis.

2. EXPERIMENTAL

2.1. Synthesis of Nickel Oxide

- Material and methods: Nickel nitrate, citric acid and glycine of analar grade are purchased from Vasa Scientific Company., Bengaluru.
- The low solution combustion technique was employed for the preparation of nanonickel oxide. 5.81 g of nickel nitrate, 2.13 g of citric acid and 1.67 g of glycine were mixed well in a magnetic stirrer for about 5 min to get uniformity in mixing and the mixture is transferred into a petri dish of 300 ml and kept in a preheated muffle furnace which is at 500°C to carry out combustion for about 8-10 min. The solution undergoes dehydration followed by decomposition with the evolution of a large amount of gases. The stoichiometry composition is calculated based on the total oxidizing and the fuel (Figure 1).
- The obtained form was grinded to get fine powder of NiO which appears in green color. The equation for combustion synthesis can be written as:

$Ni\ (NO_3)_{2(aq)} + 2C_2\ H5NO_{2(aq)} \rightarrow NiO_{(s)} +2\ N_{2(g)} +5\ H_2O +4\ CO_2$

3. CHARACTERIZATION

The XRD spectra of metal oxides we obtained by using powder XRD system Model PAN analytical source of wavelength 1.5416 Å. The FTIR spectra were recorded in KBr by using Shimadzu Model 8101 an FTIR Spectrometer. The scanning electron microscopy is made by Carl zeiss supra 55. The characteristic peaks of powders agree well whose powder peroclse (JCPDS-45-0946).

4. RESULTS AND DISCUSSION

4.1. XRD Analysis

The average crystallite size is calculated by the formula:

$$D = 0.9\lambda/FWHM(\beta)\ COS\theta\ (nm)$$

Where,

λ is the wavelength of the radiation Kα (0.1541 nm),

β is full width at half maxima (in radian),

θ is Bragg's angle. The crystallite size of nano-NiO is determined as 26.76 nm [2,3]. The high intense peak of (111) reflection shows the FCC structure. The XRD patterns indicate that the nanocomposite is well crystalline and reveals all diffraction peaks, which are perfectly similar to the literature (JCPDS no. 751526).

Percentage of crystallinity can be calculated by the formula:

(Total area of crystalline peaks/total area of all peaks)*100.

No broad peaks represent 100% crystallinity by the XRD diffractogram shown in Figure 2.

4.2. FTIR Analysis

The vibrational motion causes a change in the dipole moment of the bond. The intensity of a peak depends on the change in the dipole moment and number of the specific bonds. The intensity of the IR peaks is proportional to the change in dipole moment that a bond undergoes during vibration. In IR spectrum, the wave numbers >1400 cm^{-1} peaks represent functional group region and <1400 cm^{-1} represent finger. The middle region that lies in 4000 cm^{-1} is most useful for the analysis of IR spectra organic compound. The print region. The wave numbers between 1500 and 1000 cm^{-1} produce single bond stretch 100% transmittance shows that the sample absorbs the same

Figure 1: Solution combustion synthesis.

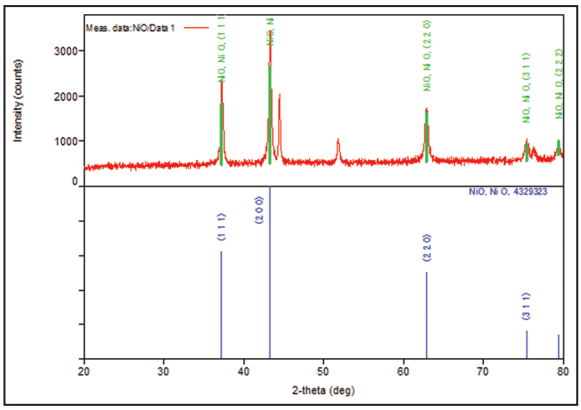

Figure 2: X-ray diffraction of NiO nanoparticles.

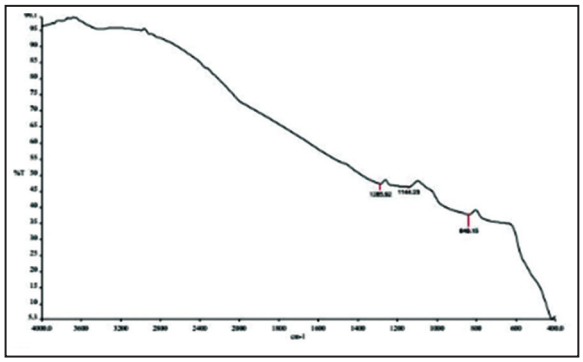

Figure 3: Fourier transform-infrared spectrum of nano NiO particles.

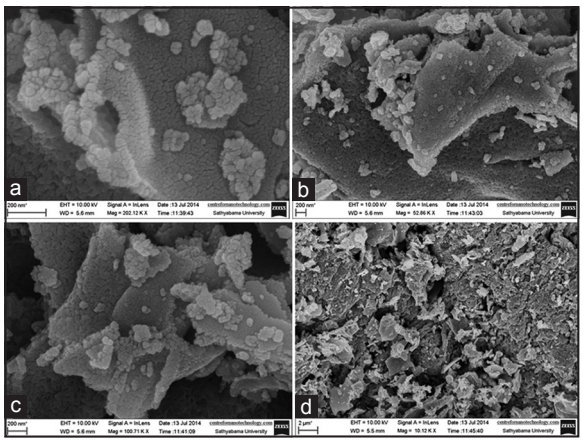

Figure 4: (a-d) Field-emission scanning electron microscopy of NiO nanoparticles.

amount of radiation as the reference. 0% transmittance means the sample absorbs all of the radiation (Figure 3).

The plot shows 100% transmittance at the top and 0% transmittance at the bottom. The FTIR transmittance peak of NiO at frequency 1285.92 cm^{-1} and 1144.23 cm^{-1} medium C-H wagging and C-H ×2 bond with alkyl halide functional group.

And also at 840.15 cm^{-1} indicates a strong C-H bond with the aromatic functional group [4].

4.3. SEM Analysis

The SEM analysis of prepared nickel oxide nanoparticles are shown in Figure 4. The Figure 4 shows that most of the particles are self-assembled and spherical in shape. Due to crystalline behavior the netting arrangement forms due to close compact [5-7].

4.4. EDXS Analysis

Chemical analysis on the nanometer scale of medium and heavy elements can be possible using EDX spectrum (Figure 5).

4.5. DC Conductivity of Nano Nickel Oxide

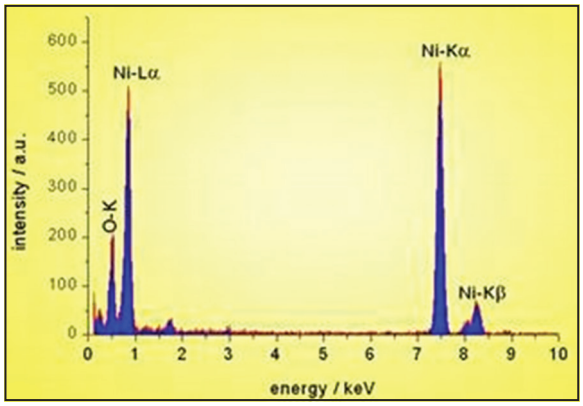

Figure 5: Energy dispersive X-ray of NiO nanoparticles.

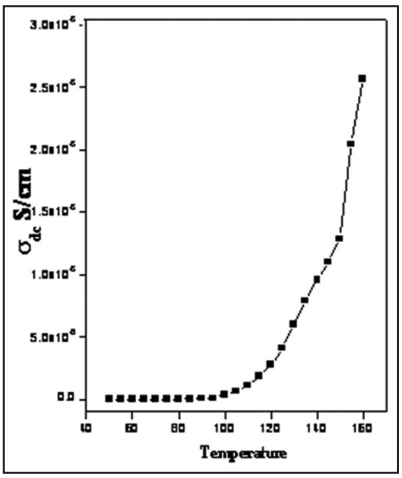

Figure 6: DC conductivity versus temperature.

The DC conductivity of the NiO sample was measured by two-probe technique in the temperature range 35-200°C. The graph plotted between temperatures versus conductivity shows the stable conductivity up to around 95°C and from that point it increases gradually and shows exponential behavior in temperature range 100-160°C. This sudden increase of conductivity after 100°C indicates the disorder behavior of semiconductor property of nano nickel oxide (Figure 6) [8].

5. CONCLUSION

The nano-sized nickel oxide is synthesized by low solution combustion method as glycine as fuel. The prepared nano-NiO particles are characterized by XRD, FTIR, SEM, and EDX. The DC electrical conductivity of the NiO sample shows the semiconductor behavior.

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****Bibliographical Sketch***

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