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Synthesis, Characterization and d.c conductivity of Nano sized Nickel Oxide

KEYWORDS: Synthesis, Carboxylate, Bonding, Structure, Morphology

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ABSTRACT *Synthesis of metal oxide materials at nano dimension integrates the materials science and technology. Decrease in particle size of the metal oxides leads to new properties and applications. Nanosized Nickel Oxide was synthesized by microwave firing through the thermal decomposition of nickel carboxylate precursor employing glycene as a fuel. This method could be an interesting because of its simplicity, eco-friendly and less time consuming. Nickel carboxylate was prepared by stirring nickel salt and carboxylic acid in a suitable solvent. This method produces nickel oxide totally free from carbon and other impurities in short time. As prepared nickel oxide is characterized for its structure by employing powder X-ray diffraction (XRD) pattern. The morphology of as prepared nickel oxide material was studied by Scanning Electron Micrograph (SEM) tool. Fourier Transform infrared (FTIR) spectral study was undertaken to know the bonding in the prepared oxide sample. The particle size and D C conductivity of the nickel oxide was studied.*

I. INTRODUCTION

Metal oxides have generated a lot of interest in recent years due to its specific properties and potential applications. These are important ceramic material of the present generation because of its high conduction and high dielectric properties [1-3]. Many researchers were attracted for the synthesis of metal oxide ceramic materials at nano dimension because of its great technological importance [4-5]. Interest has increased following the observation that the properties are strongly dependent on the size of particles with dramatic changes when nanometric sizes are acquired [6-7]. The new synthetic routes for preparation of nano structured metal oxide ceramics are under constant investigation and it finds microwave method is simple and energy efficient technique [8-9]. Irradiation of microwaves on reaction mixture converts the required product at faster rate and gives the application oriented crystalline product having controlled particle size. The oxide ceramics obtained by microwave synthetic technique shows nanocrystalline nature and also good morphology [10]. Among the various metal oxides, NiO is an important ceramic oxide used for magnetic and catalytic applications. Hence, much attention is given for the preparation, characterisation and properties of the NiO material.

Present work reports the synthesis of nickel oxide nanoparticle using nickel citrate precursor employing microwave route. Glycene is used as a fuel for the conversion of nickel citrate in to nickel oxide nanoparticles. Microwave heat treatment is given for the complete conversion of precursor in to oxide nanoparticles. The prepared sample is well characterized for its structure by X-ray diffraction (XRD), morphology by Scanning Electron Microscope (SEM) and bonding by Fourier Transform Infrared study (FT-IR) techniques. Electrical study of the prepared sample is undertaken to know its electrical behavior.

2. EXPERIMENTAL

2.1. Synthesis of Nickel Oxide

2.1.1 Materials and methods

Nickel nitrate, glycine and citric acid chemicals are used in the present study were of AR grade. Glycine is used as fuel for the combustion process and Microwave method is adopted for the synthesis of nickel oxide nanoparticles

2.1.2. Preparation of Nickel citrate precursor

The hydrated nickel citrate precursor was prepared by dissolving eqimolar proportions of Nickel nitrate and citric acid in minimum volume of solvent and was stirred for about ten minutes on a magnetic stirrer. The precipitate obtained is washed with distilled water. Finally, the precipitate is washed repeatedly with dry acetone and then dried under vaccume. [11]. As prepared nickel citrate precursor is mixed with glycine in the weight ratio 1:3 [12] and grounded well using pestle and mortar. The resultant mixture was transferred into a crucible and ignited in a microwave oven to get the desired product. Reaction mixture was placed in a domestic microwave oven having frequency 2.45GHz for about 25 minutes at 90%. The solid burns and leaving behind a light green coloured nickel oxide sample.

2.1.3. Characterization

The structures of as prepared nickel oxide were studied by X – ray diffraction using Phillips X – ray diffractometer (PW3710) with Cu Kα as source of radiation. Morphology and bonding of the above oxide was studied by Phillips XL 30 ESEM and Perkin–Elmer 1600 spectrophotometer in KBr medium tools respectively. The dc conductivity of the NiO sample were measured by four probe technique using a laboratory made setup in the temperature range 40 – 200^{o}C.

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction

Figure-1 shows indexed XRD pattern of microwave derived NiO sample. The pattern shows some peaks confirms the formation of crystalline product. The d-spacing values of the sample matches well with standard 04-0835 JCPDS file. Unit cell parameters ware obtained by least –square refinement of the powder XRD data. This study reveals that the sample is monophasic nickel oxide with cubic spinal structure having nanosized particles. The values in the parenthesis indicate miller indices

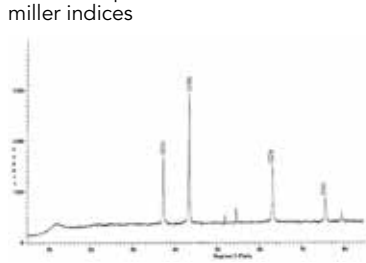

Figure-1: XRD pattern of NiO

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3.2. Scanning Electron Microscopy (SEM)

Figure-2 shows SEM image of NiO sample. This image shows, the most of the particles are self assembled spherical in shape and also a close compact arrangement forms netting due to crystalline behavior. Close mapping the particles are also observed, which enhances the properties of the materials.

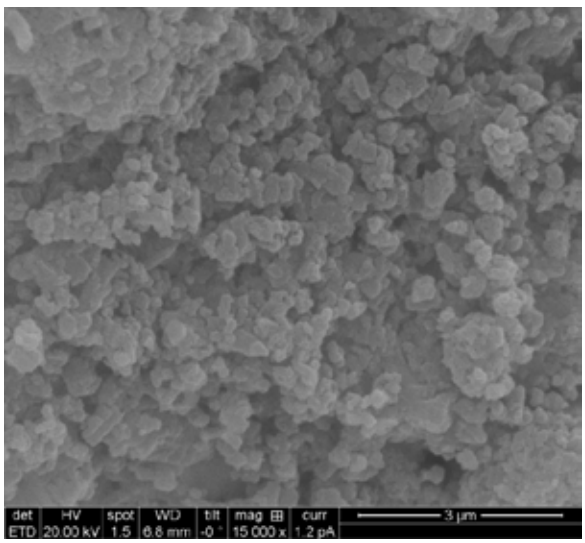

Figure-2: SEM image of NiO

3.3. Infrared Study

The aim of infrared study is to ascertain the metal- oxygen (Ni-O) bond and nature of the synthesized NiO sample. Metal oxides generally give absorption bands below 1000cm^{-1} arising from inter-atomic vibrations [13]. Observed vibrational frequencies of the said sample are given in the Table-1. The sample shows the absorption in the region 3200, 1085, 555, 500 and 440cm^{-1}. The peak 3200 cm-^{1} corresponds to water of absorption and the peak at 1085cm^{-1} due to the presence of some overtones. The peaks at 555, 500 and 440 cm^{-1} corresponds to Nickel-oxygen (Ni-O) vibrational modes of the sample conform the formation of nickel oxide.

Table-1
Vibrational Frequencies if NiO

Sample	NiO
Vibrational frequency(cm^{-1})	3200, 1085, 555, 500, 440

3.4. d. c Conductivity

Figure-3 shows the variation of dc conductivity as a function of temperature for NiO sample. The conductivity of the sample increases with increase in temperature. The dc conductivity of the said smple exhibits an exponential behavior in a temperature range 100^{0}C to 140^{0}C. In temperatures between 30^{0}–100^{0}C, the conductivity values are almost constant and increases suddenly in the temperature range 100^{0}–200^{0}C which indicates the behavior of disorder semi conductor.

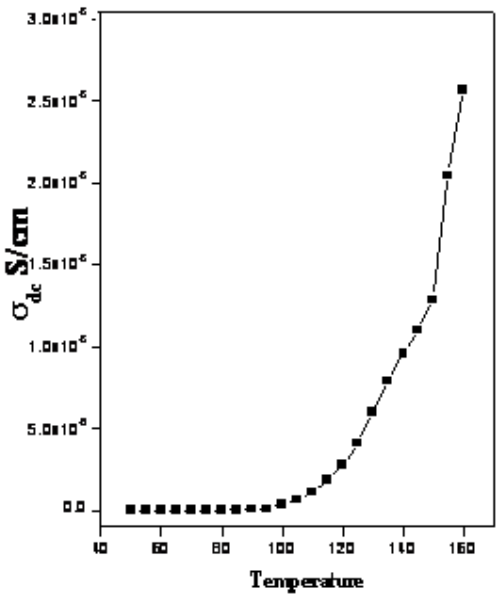

Figure-3: dc conductivity of NiO

CONCLUSIONS

The nanosized nickel oxide is synthesized by microwave irradiation with precursor and glycine as an efficient fuel. Because of its simplicity this method can adopt for the synthesis of other metal oxides at nano dimensions. This microwave preparative technique is very simple and energy efficient to obtain materials at nano dimension. Hence, this method can also adopt for the synthesis of functionalized materials at nano dimensions. The electrical study of the prepared sample shows semiconductor behavior

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