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# CHARACTERIZATION OF NICKEL OXIDE NANOPARTICLES SYNTHESIZED VIA SOLUTION-PHASE PRECURSOR ROUTE

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**Abstract:** Nickel oxide (NiO) nanoparticles were produced via a solution-phase precursor route by adding solid Guanidium carbonate in citric acid and nickel nitrate hexahydrate solution at the temperature of 95 °C in water bath. High purity NiO nanoparticles were obtained by calcinating the precipitated powders at different temperatures. Thermal analysis reveals the phase formation temperature by degrading the citrates and water content before 500°C. The strong absorption band at 419 cm<sup>-1</sup> is corresponding to the stretching vibration of NiO are confirmed by FTIR spectrum. X-ray diffraction patterns revealed that the well-crystallized/high-purity nanostructure with grain size ~35 nm of NiO nanoparticles. Scanning electron microscope and FESEM images showed that the synthesized NiO nanopowder had porous structure with pore size of ~90 nm. Nowadays porous nanopowders are using for the applications of super capacitors

## I. INTRODUCTION

Nanoparticles of transition metal oxides have attracted the attention of many materials researchers due to their exceptional properties stimulating advanced applications [1-2]. Nickel oxide (NiO) is a p-type semiconductor with a wide band gap (3.6–4.0 eV) and large exciton binding energy [3, 4]. Nanostructural NiO is considered as a promising candidate for optical, electronic, catalytic, superparamagnetic, transparent conductor film, gas sensor, alkaline battery cathode, dye-sensitized solar cell and solid oxide fuel cell anode applications [3].

Among the various preparation techniques, the solution-phase precursor route has many advantages and much promising. The advantages of this solution based technique are that it is simple, cost effective, appreciable reduction in the processing temperature and the resulting material being of nanostructure. Also, it provides excellent stoichiometry, low impurity content compared to the other methods. In this work, Guanidine metal citrate precursor was first produced by solution-phase precursor route method. Then it was post heated to form nickel oxide nanopowders. Morphological and structural characteristics of the nickel oxide were subsequently determined by SEM, FESEM and XRD.

## II. EXPERIMENTAL PROCEDURE

### Sample preparation

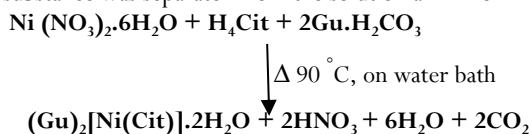
Solution-Phase Precursor Route synthesis of NiO nanopowder was composed of two stages: (a) the formation of guanidine nickel citrate precursor and (b) subsequent heat treatment of precursor to transform into NiO. To synthesis the precursor, solid

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Guanidinium carbonate was added to an aqueous solution of Citric acid with continuous stirring. To this acid-base solution an aqueous solution of Nickel nitrate hexahydrate was added. Here the metal, acid and base ratio was maintained at 1:1:4. There is a precipitate formation taking place while adding the metal to acid base solution. During the reaction the pH =9 was measured. This precipitated solution became clear with the prolonged stirring. The resulting clear solution with pH = 9 was evaporated in the water bath to reduce half of its volume. The concentrated solution was left aside over night at room temperature for re-crystallization. Finally a highly crystalline substance was separated from the solution and dried in air. Formation mechanism for metal complex,



By heating this citrate precursor at various temperature from 600 °C to 1000 °C, nickel oxide nanoparticles have been obtained.

### Results and discussions:

#### TG/DTA Analysis

As shown in Fig (i), the TG/DTA analysis confirmed that the transformation of NiO from its metal complexes taking place after 500 °C. It showed that dehydration takes place at 170 °C and removal of base followed by citrate takes place around 320-460 °C. Then the formation of NiO by thermal decomposition of the metal complex has been confirmed by the IR spectrum of the residues. The IR spectrum for NiO is shown in Fig (ii). The strong absorption band at 419 cm<sup>-1</sup> is corresponding to the stretching vibration of NiO.

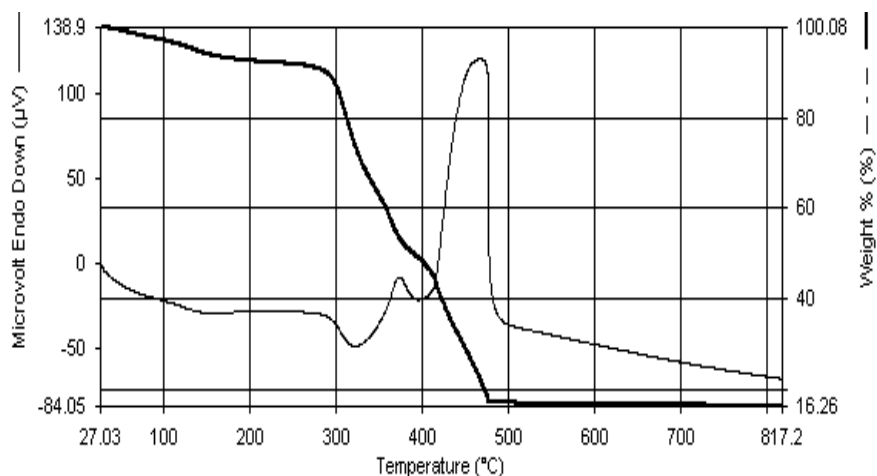


Fig (i) TG/DTA curves for the metal complex

The band at 3419 and 1383 cm<sup>-1</sup> are due to the calcinated powder tends to physically absorb water and carbonated ions [5,6].

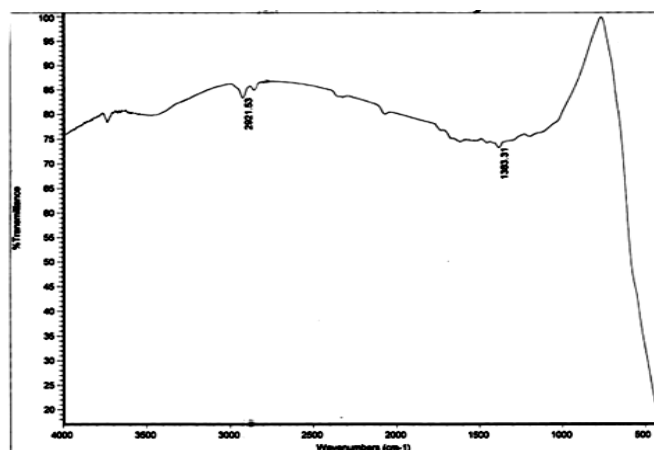


Fig (ii). IR spectrum of NiO nanopowders

The phase purity and crystallinity of the prepared samples were studied through the powder X-ray diffraction method. The powder XRD pattern of NiO nanoparticles is shown in Fig (iii). It shows that NiO nanoparticles obtained by thermal decomposition of the complex precursor with different calcinations temperatures, varied from 600 to 1000 °C at zero hour. The XRD pattern for the 600 to 800°C calcinated sample shows, few hydroxide peak in the product sample. This confirms the sample in the form of nickel

hydroxide upto 800°C calcinations. While increasing the temperature from 900 to 1000 °C the hydroxyl group disappears from the complex precursor and it form a single phase *face centered cubic (fcc)* structure of NiO. The prominent and sharp peaks of the diffractogram indicate the formation and high crystallinity of the prepared NiO. These peaks are very well match with the JCPDS # 78-0423. The average crystallite sizes of the samples were calculated by using the (111), (200), (220), (311), and (222) planes. The average crystallite size of the NiO prepared by thermal decomposition of complex precursors are calculated by using Scherrer's formula and compared with the William-Hall plot method.

$$\text{Scherrer's formula is given by, } D = \frac{K\lambda}{\beta \cos\theta}$$

Where,  $\lambda$  is the wavelength of the X-rays used,  $\theta$  is a Bragg angle,  $K$  is the geometrical shape factor and  $\beta$  be the full width half maximum of the sample in radian.

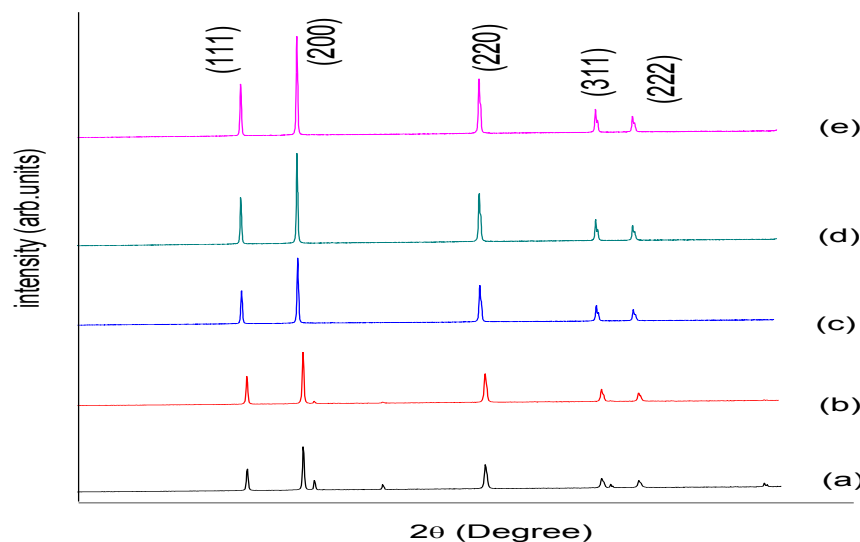
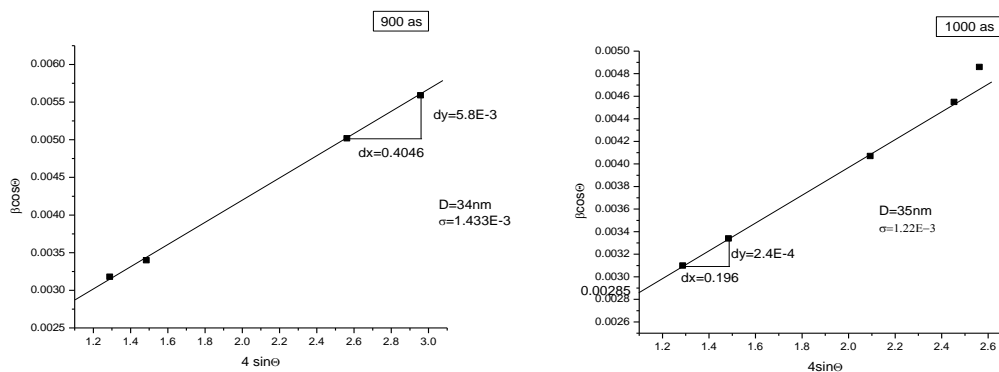


Fig (iii) Powder XRD pattern for the different calcination temperatures (a) 600 °C, (b) 700 °C, (c) 800 °C, (d) 900 °C and (e) 1000 °C



William and Hall plot for the samples prepared at 900 °C & 1000 °C

The calculated average crystallite sizes, strain and dislocation density were tabulated.

Samples calcination temperatures (°C)	Avg. Grain size calculated from (nm)		Strain $\text{lin}^{-2}\text{m}^{-4}$	Dislocation Density $\text{lines}/\text{m}^2$
	Scherrer formula	W-H plot method		
900	36	34	1.43E-03	7.70E-04
1000	37	35	1.22E-03	7.30E-04

In both cases the increase in the calcination temperature the reduction in the lattice strain is observed.

The SEM images for the samples calcinated at different temperatures are shown in fig (iv).

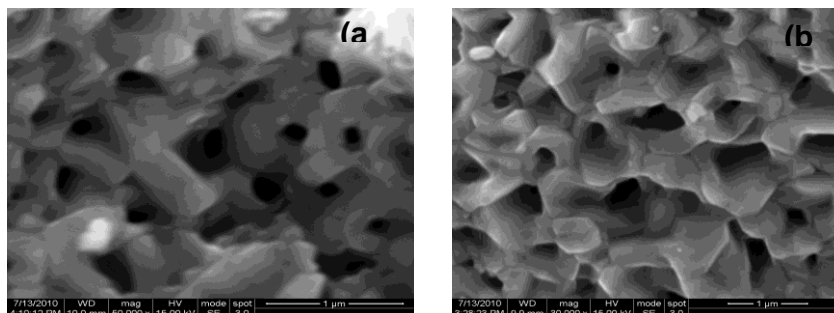


Fig (iv) SEM image for the calcination temperatures of (a) 900 °C and (b)1000°C

While increasing the calcinations temperature, the clear and smooth surface morphologies were obtained. Due to their nanoscale size and surface properties, also nanoparticles tend to aggregate or precipitate in suspensions. The increase in the calcination temperature leads to the formation of porous like structure in the morphology [7]. Further increase in the calcination temperature also yields smooth porous like morphologies. The morphology of the 1000 °C calcinated sample shows the piled up plates. This particular morphology is considered as an optimized sample for further analysis. Based on the scanning electron micrographs, the pore size histograms can be drawn and mean pore size can be determined. Fig (v) shows the pore size distribution for NiO nanoparticles at 1000 °C. The average pore size of the 1000 °C sample is in the range 90 to 300 nm.

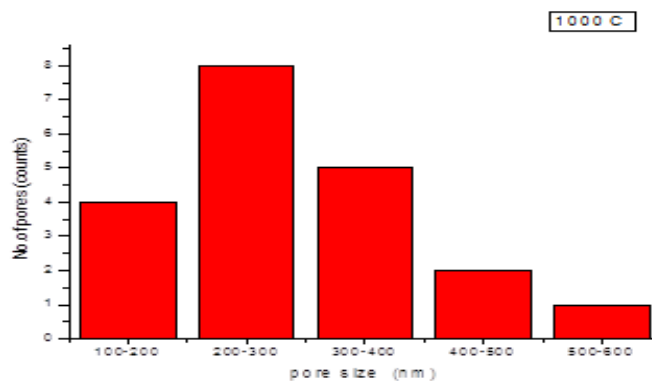


Fig (v) Pore size distribution of 1000 °C calcinated sample

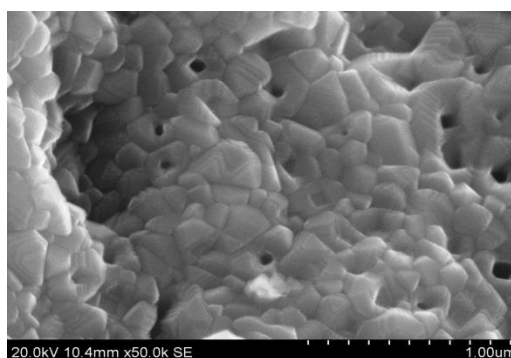


Fig (vi) FESEM image of 1000°C Calcinated sample in air.

The FESEM image of the sample is shown in fig (vi). It shows a fascinating structure for the optimized sample. The image shows the triangle like plates are piled up and the particles are in uniform in size with the prism like grains which are well connected to provide porous structure which is much required for device applications, for instance super capacitors. Since electric double-layer capacitance and pseudocapacitance are interfacial phenomena, the materials for electrochemical capacitors should possess a high specific surface area with a suitable pore-size distribution to enhance the charge-storage capability. In this work we have developed a mesoporous Nickel Oxide with a very high surface area is developed by an anionic template.

#### Conclusion:

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Highly-crystallized porous nickel-oxide nanoparticles with average crystallite size of ~35nm were synthesized by a solution-phase precursor route method. Morphology of the resulted powder showed triangle like plates are piled up with lots of pores on the surface. This method resulted in formation of a well-shaped crystalline and mesoporous product. This phenomenon could be attributed to stoichiometry of the nanoparticles obtained.

**References:**

- [1]. P. Duran, J. Tartaj, C. Moure, J. Am. Ceram. Soc., Vol. 86 (8), 2003, pp. 1326–1329.
- [2]. M. Mazaheri, A.M. Zahedi, M.M. Hejazi, Mater. Sci. Eng. A, Vol. 492, 2008, pp. 261–267.
- [3]. J. Bahadur, D. Sen, S. Mazumder, S. Ramanathan, J. Sol. Sta. Chem., Vol. 181, 2008, pp. 1227–1235.
- [4]. H. Sato, T. Minami, S. Takata, T. Yamada, Thin Solid Films, Vol. 236, 1993, pp. 27.
- [5]. Hongxia Qiao, Zhiqiang wei, Hua yang, Lin Zhu, and Xiaoyan yan, journal of Nanomaterials. Volume 2009.
- [6]. Y. Bahari Molla Mahaleh, S.K.sadrnezhaad, and D. Hosseini, Journal of nanomaterials, volume 2008.