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Synthesis and Characterization of NiO Nanoparticles by Non-Aqueous Sol-Gel Route

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***Abstract**: In the present work nickel oxide nanoparticles (NiO) were successfully synthesized by non-aqueous sol gel route, in presence of potassium hydroxide (KOH). The nanoparticles are uniformly distributed in modifiers in Non-aqueous sol-gel method in large area, which is due to an extremely high stability of charged nanoparticles generated from $Ni(NO_3)_2$/KOH solution. The particles are Spherical in the case of $Ni(NO_3)_2$/KOH ZnO nanoparticles, and it is decreased in higher concentration. The conclusions are drawn with the help of various instrumental techniques like scanning electron microscope (SEM), and transmission electron microscope (TEM) and X-Ray Differectometer (XRD).*

Keywords: Nanoparticles, Sol-gel method, Scanning electron microscope (SEM), Transmission electron microscope (TEM), Nickel oxide

I. INTRODUCTION

Nickel oxide nanoparticles can be manufactured by thermal decomposition of freshly prepared nickel oxide by sol gel route at 300°C (572°F). The nickel oxide nanoparticles created using this method can be characterized using x-ray diffra ctometer and vibrating sample magnetometer. NiO adopts the NaCl structure, with octahedralNi(II) and O^{2-} sites. . NiO is a versatile hydrogenation catalyst. Heating nickel oxide with either hydrogen, carbon, or carbon monoxide reduces it to metallic nickel. It combines with the oxides of sodium and potassium at high temperatures (>700 °C) to form the corresponding nickelate.

Nickel is a Block d, Period 4 element, while oxygen is a Block P, Period 2 element. Nickel oxide nanoparticles appear in green powder form, and are graded as very toxic. Nickel(II) oxide is the chemical compound with the formula NiO. It is notable as being the only well characterized oxide of nickel (although nickel(III) oxide, Ni_2O_3 and NiO_2 have been claimed. The mineralogical form of NiO, bunsenite, is very rare. It is classified as a basic metal oxide. Several million kilograms are produced in varying quality annually, mainly as an intermediate in the production of nickel alloys. They can cause an allergic skin reaction, prolonged harmful effects to aquatic life, and possible damage to organs due to prolonged or repeated exposure. NiO was also a component in the Nickel-iron battery, also known as the Edison Battery, and is a component in fuel cells. It is the precursor to many nickel salts, for use as specialty chemicals and catalysts. More recently, NiO was used to make the NiCd rechargeable batteries found in many electronic devices until the development of the environmentally superior Lithium Ion battery. About 4000 tons of chemical gradeNiO are produced annually. Black NiO is the precursor to nickel salts, which arise by treatment with mineral acids. NiO is a versatile hydrogenation catalyst. Heating nickel oxide with either hydrogen, carbon, or carbon monoxide reduces it to metallic nickel. It combines with the oxides of sodium and potassium at high temperatures (>700 °C) to form the corresponding nickelate.

II. MATERIALS AND METHOD

A. Materials

Nickel nitrate [Ni $(NO_3)_2$, Merck, AR grade, 182.703 g/molDensity: 2.05 g/cm³), Potassium hydroxide (KOH, Rankem, AR grade, 56.1056 g/mol, Density: 2.04 g/cm³], Oxalic acid (merck, AR grade,90.03 g/mol, $C_2H_2O_4.2H_2O$,Density:1.90g/cm³), Ethanol (CH_3CH_2OH, Rankem, AR grade, 46.07 g mol^{-1}, Density: 0.789 g/cm^3), p^H strips (Baker-pHIX p^H Indicator Strips), Whatman quantitative filter paper (sigmaaldrich, ashless, Grade 42 circles, diam. 42.5 mm, pack of 100), Glycerol (Merck, 1,2,3-Propanetriol, Density-1.23 g/cm^3).

B. Method

1) Step-1 Preparation of $Ni(NO_3)_2$ and KOH solution: Then made this solution carefully in 100 ml volumetric flask.

1.402 gram of KOH was dissolved in 10 ml of ethanol in RB flask and stirred it for 10 minutes until it was dissolved. Then made this solution carefully in 25 ml volumetric flask.

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2) *Step-2 Synthesis of NiO nanoparticles:* Pour the 0.2 M nickel nitrate solution in RB flask and fix it on magnetic stirrer. The sol was prepared by adding 1 M KOH drop by drop in nickel nitrate solution, until the pH of solution reaches to 9. Stirred it for 3 hours. For gelatin added 1 ml glycerol in $Ni(NO_3)_2$ and KOH solution then stirred it for 30 minutes. Kept overnight for setting down the particles. Next day filtered the NiO nanoparticles and wash them with 1:1 alcohol/water ratio solution 2 to 3 times then dried the particles in hot air oven at 80^{o}c for 3 hours. After the calcinations at 450^{o}c in muffle furnace pure NiO nanoparticles were obtained.

III.RESULT AND DISCUSSION

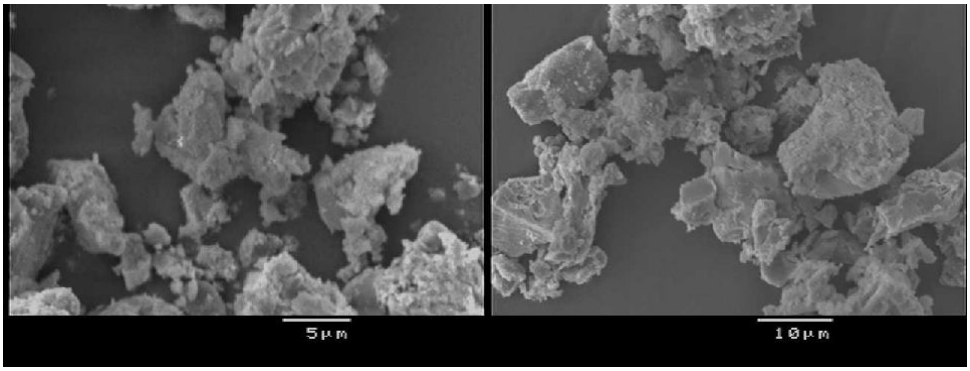
Nickel oxide nanoparticles were synthesized by non aqueous sol-gel route to study the effect on particle size and agglomeration of nanoparticles in the presence of oxalic acid and potassium hydroxide used in different molar ratio to control the particle size and agglomeration of nanoparticles. The conclusion are drawn with the help of various instrumental techniques like scanning electron microscope (SEM), X-ray diffraction (XRD) and transmission electron microscope (TEM). Below figure shows the NiO nanoparticles before and after the calcinations.

A B

NiO nanoparticles (A) Before calcination and (B) After calcination

A. SEM Analysis

The SEM micrographs of nickel oxide nanoparticles having different ratio of $(NiNO_3)_2$/KOH.The microstructure of the NiO nanoparticles synthesized by Non aqueous sol-gel method in the present study was observed in the presence of KOH to control the particle size of NiO nanoparticles by SEM. From the SEM images it is clear that the NiO nanoparticles are well dissolved in ethanol and KOH solution. As the concentration of nickel nitrate and KOH increases nanoparticles are found to be agglomerated as shown in figure.



SEM images of NiO nanoparticles in the presence of KOH

B. TEM Analysis

To determine the exact particle size and distribution TEM was done. TEM images were recorded using JEOL 3010 electron microscope. The sample for TEM were prepared by putting a powder NiO nanoparticles over the carbon supported copper grid. The samples were vacuum dried before putting them in a specimen holder. TEM images containing different concentration of nickel nitrate and KOH are shown in figure. TEM analysis shows that the particles are of narrow shape and are of nanometre size. As the concentration increased the particles were found to be much agglomerated shown in figure. This was also supported by XRD of NiO

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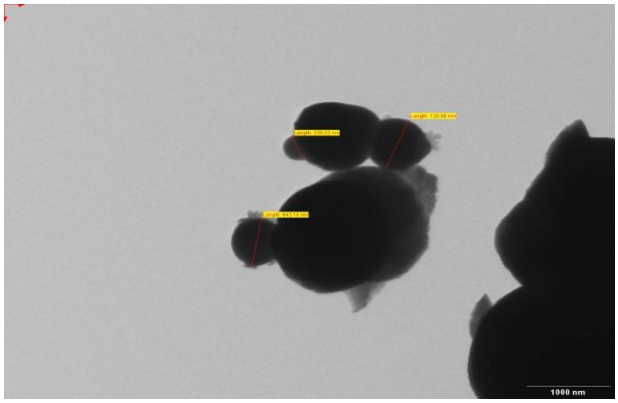
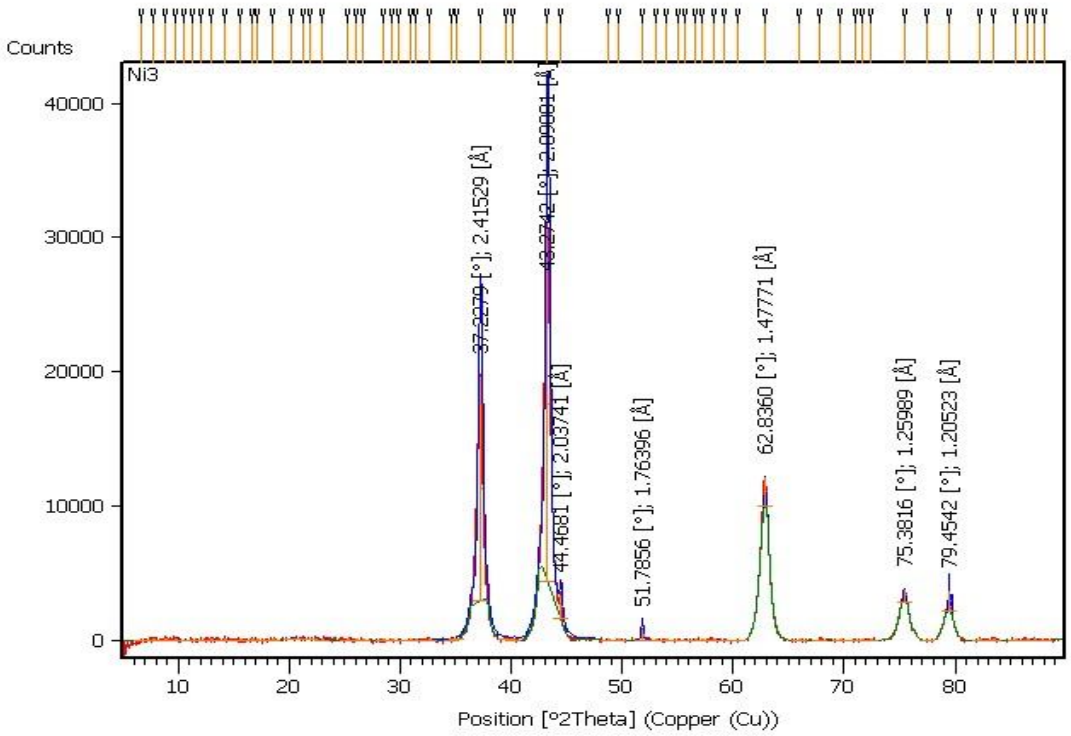
nanoparticles having different concentration of nickel oxide nanoparticles. The average diameter is found to be around 329 nm of NiO nanoparticles.



TEM image of NiO nanoparticles

C. X-Ray Diffraction

The X-ray diffraction spectrum of the calcined powder was recorded using Instrument is panalytical and empyrean respectively Used target is Cu K-alpha with the wavelength of target is 1.54 Angstroms (0.154 nm). It is well known that the calcinations improve the crystallinity of the particle, and the NiO changes to the cubic phase. The particle calcined at 700^{o}C.



XRD pattern of NiO nanoparticles

The crystal phase of the sample was analysed by X-ray diffraction (XRD). The powder XRD pattern of the sample in our experiment is shown in Fig. The characteristic peaks are at 2θ = 37.22°, 43.27° and 62.83° [corresponding to (111), (200) and (220) reflection, respectively]. The sample is phase-pure NiO, all the identified peaks of which can be assigned to the cubic phase of NiO. The peaks have been identified as peaks of cubic NiO crystallites with various diffracting planes.Their broadening could be attributed either to their micro-structural distortion. Under the present experimental conditions, the extremely small particle-like

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structure could be more motivation for a significant broadening of some diffraction peaks in NiO XRD patterns. All of these diffraction peaks in Figure, not only the peak positions appearing at 2θ= 37.28, 43.28, 62.88, 75.28, and 79.48 but also their lattice parameters, were quite consistent with those of the standard JCPDS Card No. 04-0835 for the standard spectrum of the pure and cubic NiO. It was seen that these characteristic diffraction peaks in the pattern had a marked broadening effect. The results indicated that the products were nano-NiO crystal of cubic structure; they have a high purity and small particle size with a fine crystal phase.

IV.CONCLUSION

Nickel oxide nanoparticles were synthesized through sol-gel method. The results of the TEM and XRD showed that the average particle size of NiO Nanoparticles increases with increasing concentration of potassium hydroxide solution, and the SEM results showed that the formation of narrower shaped nanoparticles.

V. ACKNOWLEDGEMENT

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CONCLUSIONS

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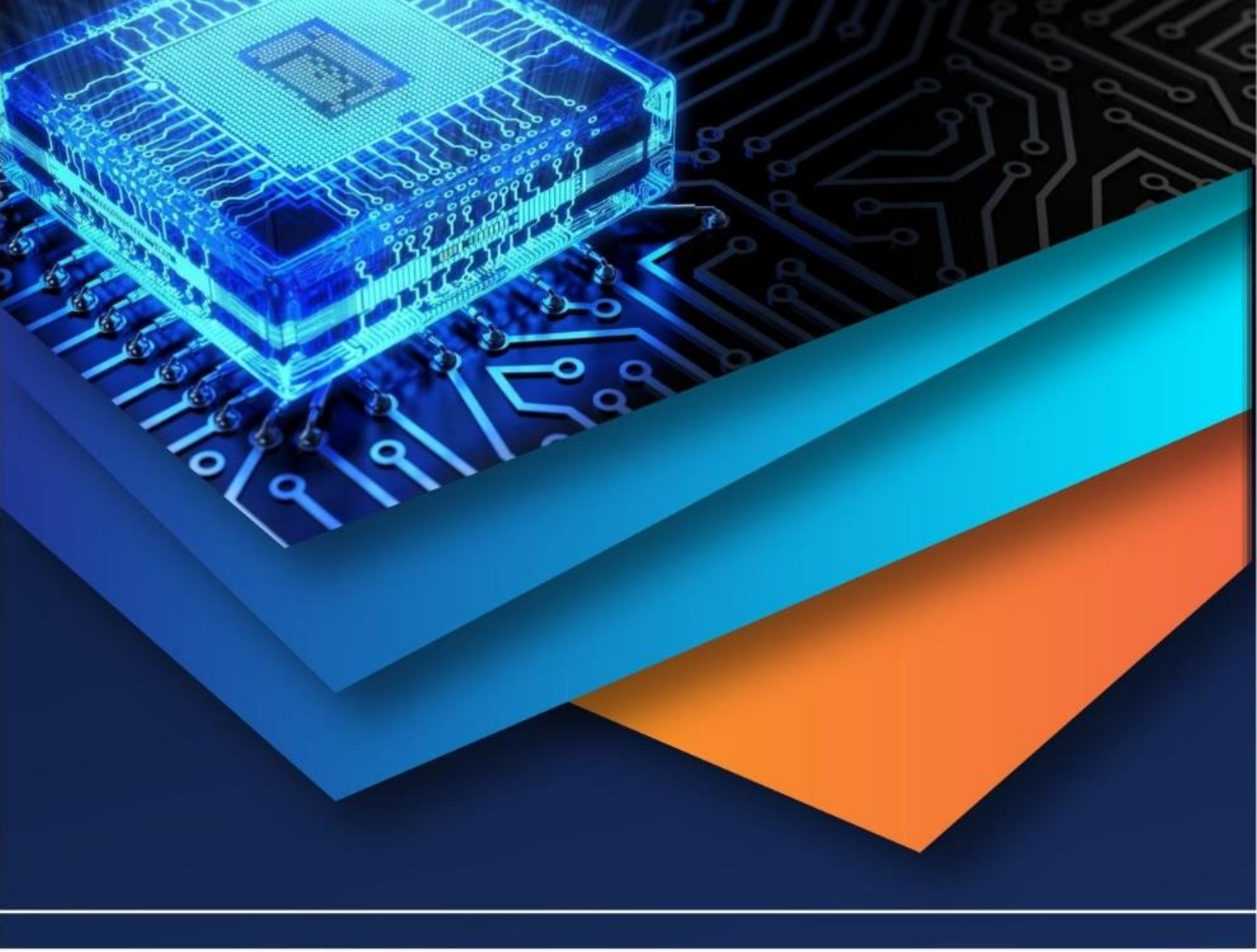

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