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Synthesis and Characterization of NiO Nanopowder by Sol-Gel Process

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Abstract. Preparation of nickel oxide (NiO) nanopowder by sol-gel process has been studied. NiO nanopowders were obtained by sol-gel method by using nickel nitrate hexahydrate and sodium hydroxide and aquadest were used as precursor, agent precipitator and solvent, respectively. The powders were formed by drying at 110 ^{o}C and followed by heating in the furnace at 400 ^{o}C for 1.5 hours. The product was obtained black powder. The product was characterized by Energy Dispesive X-ray Fluorescence (ED-XRF), X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). The ED-XRF pattern shows the composition of NiO produced was 97.1%. The XRD pattern showed NiO forms were produced generally in monoclinic stucture. The crystalline size of NiO was obtained in the range 40-85 nm. SEM micrograph clearly showed that powder had a spherical with uniform distribution size is 0.1-1.0 μm approximately.

Keywords: *nickel oxide, precursor, solvent, sol-gel, monoclinic, spherical*.

INTRODUCTION

Nanoparticles are classified as being materials in which at least one dimension of the material is less than 100 nanometers in diameter. Nanoparticles have unique properties, such as having increasing electrical conductivity, toughness, ductility and formability of ceramics. The research of diffrent types of the nanoparticles has been an active in the last decade.

Nickel oxide (NiO) is kind of a p-type semoconductor that can be used in optical, anti-ferro-magnetic film[1], electronic, catalytic, superparamagetic device [2], supercapacitors [3], gas sensors, alkaline batteries cathode, solid oxide fuel cell anode and in the manufacture of electrochromic [4]. NiO has a large exciton binding energy and a wide band gap ranging from 3.6 to 4.0 eV [2]. NiO nanoparticles show many unique optical, electrical, magnetic and chemical properties [5].

There are many techniques that are closely related to the particle structural property such as partice size, distribution particle and morphology. Nanoparticle NiO can be prepared by many methods including thermal treatment, chemical precipitation and solvothermal method [4,6], soft chemical synthesis route [7] involved solid liquid seperation, washing and drying process., microwave irradiation method [1-2], liquid phase process, spray pirolysis process, electroplating of nickel particle [3], metal organic chemical vapor deposition (MOCVD) [5] has a number disadvantages such as quite expensive, uses volatile compound and need complicated reactor.

In this work, we synthesized nanopowder NiO by sol-gel method by using nickel nitrate hexahydrate as precursor, aquadest as solvent and sodium hydroxide 5 M as additive or agent precipitation. Sol-gel process has a number of

advantages over other methods such as inexpensive equipment, homogenous product and easily in processing. Sol-gel method will produce small particle size and uniform distribution particle with high homogenous.

MATERIAL AND METHODS

All the chemical reagents used in this experiment were of analytical grade and without further purification. Powders NiO synthesized by sol-gel method with solvents variation by using nickel nitrate hexahydrate from Merck as precursor, aquadest as solvent, sodium hydroxide as agent precipitator. 5.0 M NaOH was added drop wise 20 mL of 2.0 M $Ni(NO_3)_2.6H_2O$ solution which was stirred by magnetic stirring apparatus, keep stirring the solution for ± 2 hours at room temperature. Light green suspension was produced. It was then dried at 110 ^{o}C for 1 hour, the green powder was produced. The product then transferred to porcelain crucible and put in furnace at 400 ^{o}C for 1.5 hours, and finally black powder produced.

To identify and the structure of the synthesized NiO nanoparticle was characterized by using X-ray Diffraction (XRD). The XRD was performed by X'Pert Pro MRD from PAnalytical with CUKα radiation λ = 0.15406 nm.the composition of NiO sample was analyses by using Energy Dispersive X-Ray Fluorescence (ED-XRF). The morphology of NiO sample was carried out by using Scanning Electron Microscopy (SEM).

RESULTS AND DISCUSSION

NiO nanopowder was synthesized by using sol-gel process. The precursor was nickel nitrate hexahydrate, aquadest as solvent. The NaOH 5M was added to the solution of NiO as precipitation agent.
The reaction can be seen below:

$2 NaOH_{(aq)} + Ni(NO_3)_2.6H_2O_{(aq)} \rightarrow 2NaNO_{3(aq)} + Ni(OH)_2.6H_2O_{(aq)}$
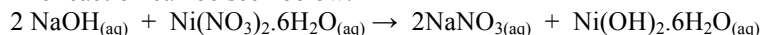
Nickel nitrate hexahydrate was easily dissolved in aquadest solvent. The sol of NiO was in green color. After addition of sodium hydroxide solution, the sol of NiO changed to green precipitation (Figure 1). To remove the solvent, the gel of NiO was heated in oven at 110 ^{o}C for 1 hour. The aquadest solvent was more evaporate.
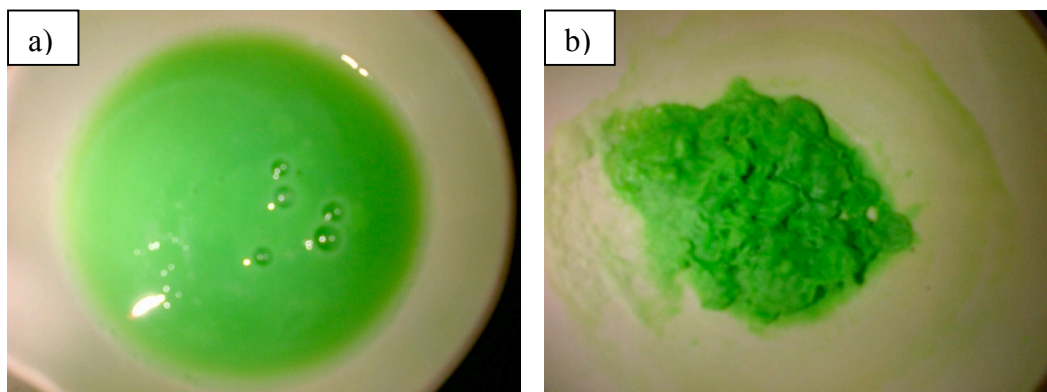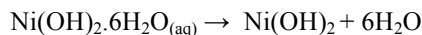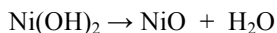The reaction can be seen below:

$Ni(OH)_2.6H_2O_{(aq)} \rightarrow Ni(OH)_2 + 6H_2O$

FIGURE 1. a) sol of NiO and b) gel of $Ni(OH)_2.6H_2O$ (after drying at 100-110 ^{o}C)

The gel of NiO was calcined at 400 ^{o}C for 1.5 hours in furnace. The reaction can be seen below, NiO was produced after calcination at 400 ^{o}C in furnace, the reaction below:
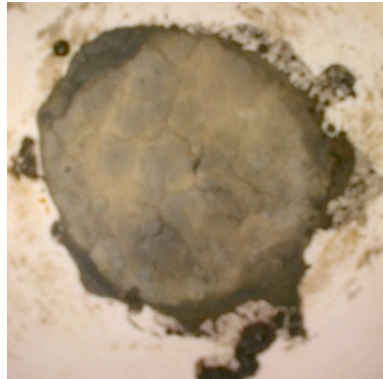
$$Ni(OH)_2 \rightarrow NiO + H_2O$$

FIGURE 1. NiO powder (after calcination process at 400 ^{o}C)

Figure. 2 shows the NiO powder after calcination in furnace at 400 ^{o}C for 1.5 hours. The black powder produced. The powder in visual was homogenous, light black color of NiO, the particle size was very small.

Figure 3 shows the ED-XRF pattern of NiO powder by sol-gel process. From this figure can be seen that composition of NiO is 97.1 %, Al_2O_3 is 0.7 %, P_2O_5 is 0.9%, CaO is 0.1 %. From this curve showed that composition of NiO is highest. It means the purity of NiO was very high (Table 1).

TABLE 1. XRF data of NiO nanopowder

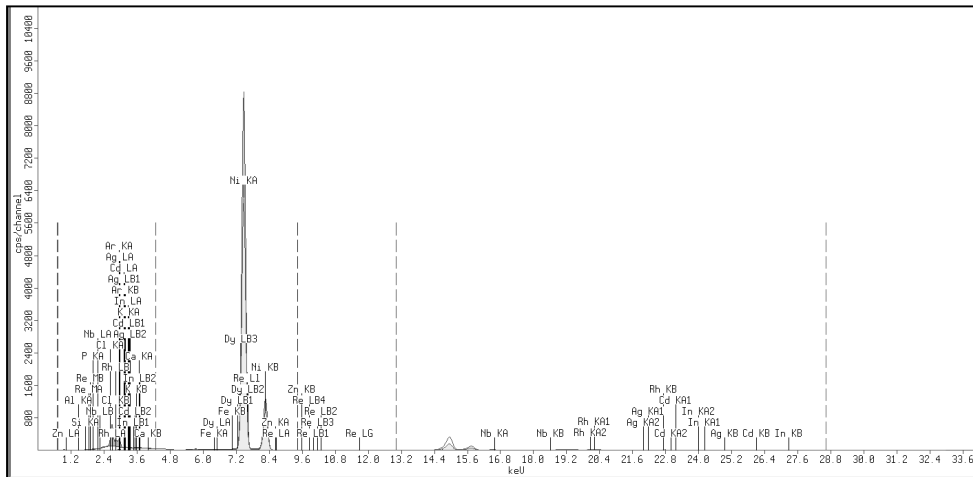| Compound	Concentration	Unit
Al_2O_3	0.639	%
SiO_2	0.702	%
P_2O_5	0.872	%
Cl	0.291	%
K2O	0.042	%
Limestone	0.251	%
Hermatite	0.028	%
NiO	96.81	%
ZnO	0.023	%
NbO_2	0	%
Ag_2O	0	%
CdO	0.274	%
In_2O_3	0	%
Dy	0.07	%
ReO_4	0.003	%

FIGURE 3. ED-XRF pattern of NiO at 400°C with sol-gel process

Figure 4 shows the X-ray diffraction pattern of the black powder of NiO prepared by sol-gel process. The data clearly depicts at 2θ= 37.22; 43.25; 47.87; 62.83; 63.00 and 75.37. The XRD pattern revealed the formation of monoclinic structure of NiO (Pdf Card No. 01-072-4521). The other peaks observed at 2θ= 29.34 and 31.83 which have been identified as sodium nitrate peak (Pdf Card No. 01-079-2056). The crystalline size of NiO nanopowder by using the the Scherrer formula vary in the range of 40-85 nm (Table 2).
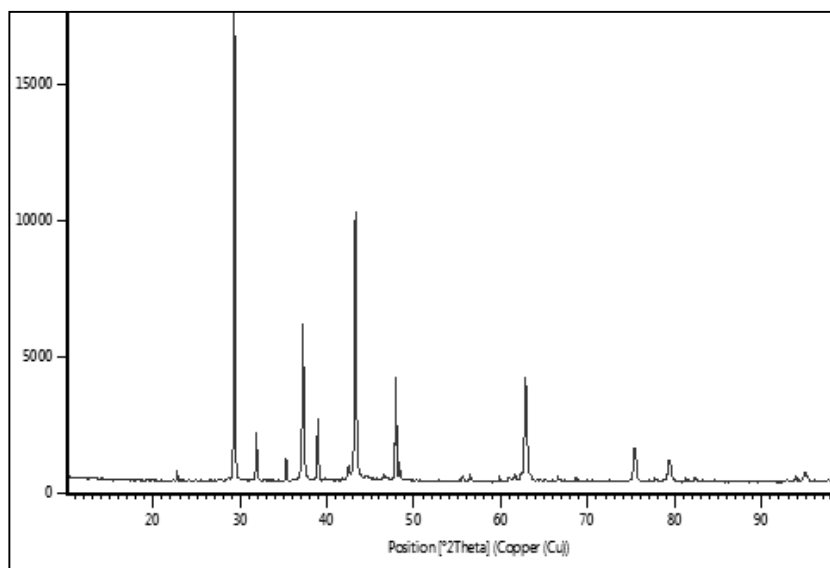

FIGURE 4. X-ray diffraction pattern of NiO nanopowder

TABLE 2. FWHM and crystalline size of NiO nanopowder

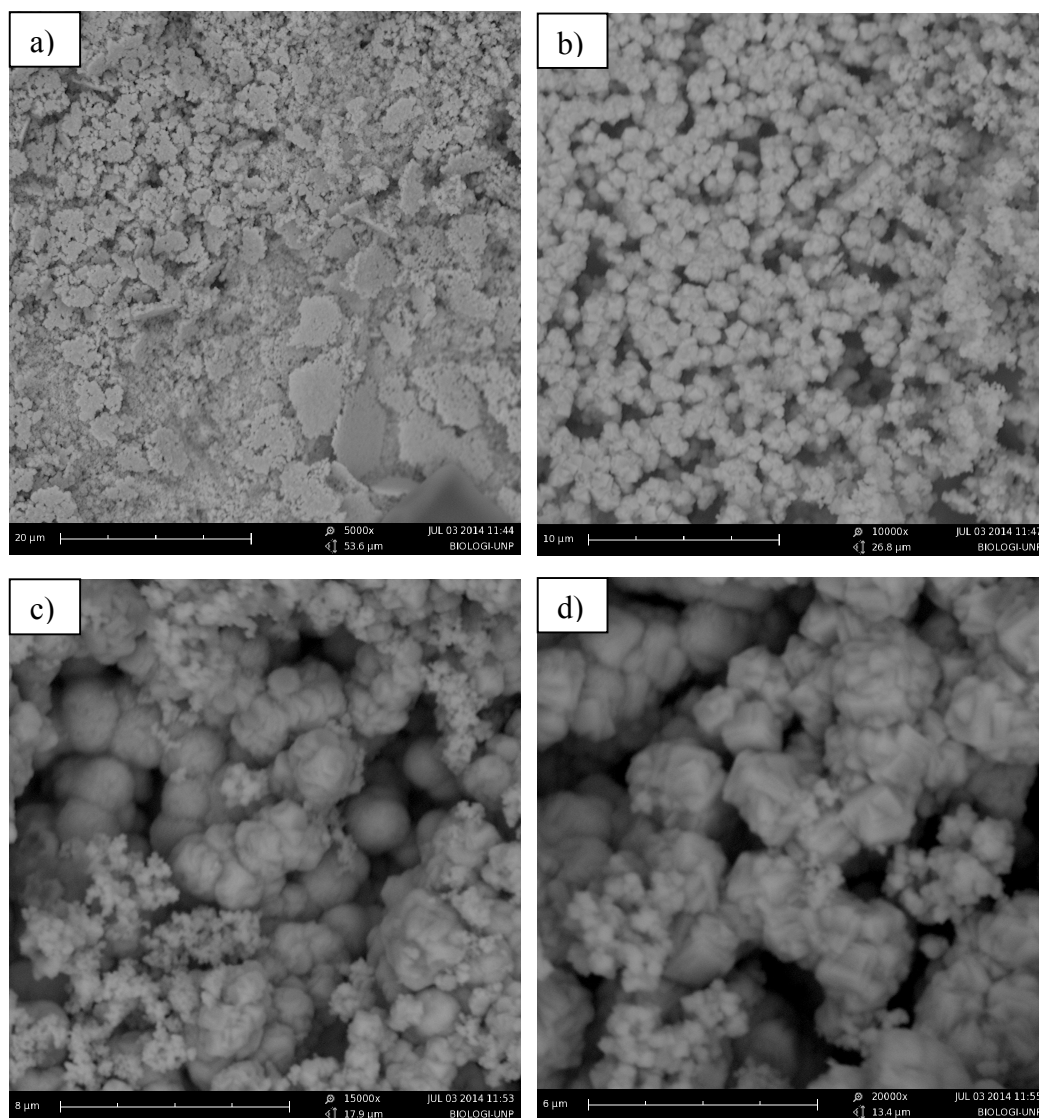| Angle (2θ)	Height (counts)	FWHM	d-value([Å]	Relative intensity (counts) (%)	Crystallite size (nm)
37.22	5422.68	0.2047	2.4156	44.52	40.3
43.25	9017.30	0.2047	2.0920	74.03	41.5
47.87	1682.76	0.1023	1.9003	13.82	85.7
62.83	3504.30	0.2184	1.4778	28.77	42.2
63.00	2305.27	0.1248	1.4777	18.93	72.2
75.37	1192.16	0.2184	1.2600	9.79	45.7

FIGURE 5. SEM Micrograph of NiO at 400 ^{o}C for 1,5 hours (a) 5,000x, (b) 10000x, (c) 15,000x, and (d) 20,000x

Figure 5 depicts the SEM image NiO nanopowder that characterized by using Scanning Electron Microscopy. The SEM micrograph clearly showed that NiO powder had a spherical with uniform distribution size is 0.1-1.0 μm approximately. From the SEM analysis, the microstructure of NiO nanopowder was produced in spherical form. The particle size of NiO nanopowder was small and its can be uses as catalyst and adsorbent. From the SEM micrograph can be seen that the agglomeration of NiO nanopowder.

CONCLUSION

NiO nanopowder was succesfully synthesized by sol-gel process. NiO nanopowder prepared by nickel nitrate hexahydrate 2M as precursor, aquadest as a solvent and sodium hydroxide 5M as agent precipitation. The NiO nanopowder was heated in furnace at 400 ^{o}C for 1.5 hours. The product was black powder. The crystalline size of NiO nanopowder was calculated by Scherrer Formula. The crystallite size of NiO nanoparticle was 37.05 nm. The structure of NiO nanopowder with aquadest solvent was a monoclinic type. Micrograph of NiO nanopowder was spherical form with diameter size in range 0.1-1.0 μm approximately.

ACKNOWLEDGMENTS

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