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Nickel Oxide (NiO) nanoparticles prepared by solid-state thermal decomposition of Nickel (II) schiff base precursor

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Abstract
In this paper, plate-like NiO nanoparticles were prepared by one-pot solid-state thermal decomposition of nickel (II) Schiff base complex as new precursor. First, the nickel (II) Schiff base precursor was prepared by solid-state grinding using nickel (II) nitrate hexahydrate, $Ni(NO_3)_2{\cdot}6H_2O$, and the Schiff base ligand *N,N′*-bis-(salicylidene) benzene-1,4-diamine) for 30 min without using any solvent, catalyst, template or surfactant. It was characterized by Fourier Transform Infrared spectroscopy (FT-IR) and elemental analysis (CHN). The resultant solid was subsequently annealed in the electrical furnace at 450 °C for 3 h in air atmosphere. Nanoparticles of NiO were produced and characterized by X-ray powder diffraction (XRD) at 2θ degree 0-140°, FT-IR spectroscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The XRD and FT-IR results showed that the product is pure and has good crystallinity with cubic structure because no characteristic peaks of impurity were observed, while the SEM and TEM results showed that the obtained product is tiny, aggregated with plate-like shape, narrow size distribution with an average size between 10-40 nm. Results show that the solid state thermal decomposition method is simple, environmentally friendly, safe and suitable for preparation of NiO nanoparticles. This method can also be used to synthesize nanoparticles of other metal oxides.

Keywords: *Nickel (II) Schiff base precursor, NiO nanoparticles, prepared, SEM, TEM, XRD.*

1. Introduction

In recent years, nickel oxide nanoparticles have been extensively investigated due to their properties [1] and potential applications in many fields [2]. Nickel oxide is also an interesting anode material for lithium-ion battery [3,4] owing to its high chemical and thermal stability and low cost [5]. Many research scientists are working toward the synthesis of high quality NiO nanoparticles using various methods [1-8]. However, most of them are still limited by laboratory, low-yield and high-cost. It is essential to develop low-cost method for the preparation of NiO. Among the all methods used for the

preparation of NiO nanoparticles, solid-state thermal decomposition [9-14] of a suitable precursor exhibits many advantages; which are simplicity and low-cost, because it does not need complex apparatus, it is environmentally friendly, and there is no need for solvent and surfactant. This method can be used to synthesize nanoparticles of other metal oxides. Recently, a wide variety of transition metal oxide nanoparticles have been synthesized by this method, including CuO [15-17], Mn_3O_4 [18, 19] and Co_3O_4 [16, 20].

Up till now, nickel oxide powders with various structures such as spherical, hexagonal, octahedral, flower and tube have been synthesized and characterized [1-14]. Previous works [11, 12, 16, 21-23], have proposed the preparation of NiO nanostructures from solid-state thermal decomposition of new nickel (II) Schiff base precursor prepared by solid-state grinding using $Ni(NO_3)_2{\cdot}6H_2O$ and the Schiff base ligand *N*,*N*′-bis-(salicylidene) benzene-1,4-diamine).

2. Experimental

2.1. Materials and physical measurements

All reagents and solvents for synthesis and analysis were commercially available and used as received without further purifications. *N*,*N*′-bis-(salicylidene) benzene-1,4-diamine was prepared according to the method of Kondo et al. [24]. X-ray powder diffraction (XRD) pattern of the complex was recorded on a Bruker AXS diffractometer D8 ADVANCE with Cu-Kα radiation with nickel beta filter in the range $2\theta = 10^o - 80^o$. Fourier Transform Infrared spectra were recorded as a KBr disk on a FT-IR Perkin Elmer spectrophotometer. The transmission electron microscopy (TEM) images were obtained from a JEOL JEM 1400 transmission electron microscope with an accelerating voltage of 120 kV while the scanning electron microscopy (SEM) images were obtained from a Philips XL-30ESEM.

2.2. Preparation of NiO nanoparticles

$Ni(NO_3)_2{\cdot}6H_2O$ (1 mmol) and Schiff base ligand (1 mmol) were grinded separately for 5 min in an agate mortar. The powders were then mixed and grinded for 30 min. The product was washed with ethanol and dried at 70 °C in an oven. FT-IR (KBr, cm^{-1}): 1609 (C=N).

The resultant solid was subsequently annealed in an electric furnace at 450 °C for 3 h. Nanoparticles of NiO were produced after 3 h, washed with ethanol and dried at room temperature. FT-IR (KBr, cm^{-1}): 3446 (H_2O), 418 (Ni-O).

3. Results and Discussion

3.1. FT-IR spectra

In the FT-IR spectrum (Fig. 1) of nickel precursor, the absorption bands at about 3000 cm^{-1} and 1400-1550 cm^{-1} are attributed to the C-H and C=C stretching vibration of aromatic protons and carbons from corresponding ligand, respectively. The absorption band that appeared at 1609 cm^{-1} is attributed to the C=N stretching vibration of ligand [24]. As can be seen in Figure 1, all bands of the precursor had disappeared and a strong band at 418 cm^{-1} was observed which was assigned to the Ni-O stretching [9-14]. Also, the two bands which appeared at about 3446 and 1630 cm^{-1} are related to the water molecules absorbed by the NiO nanoparticles or KBr [9-14].

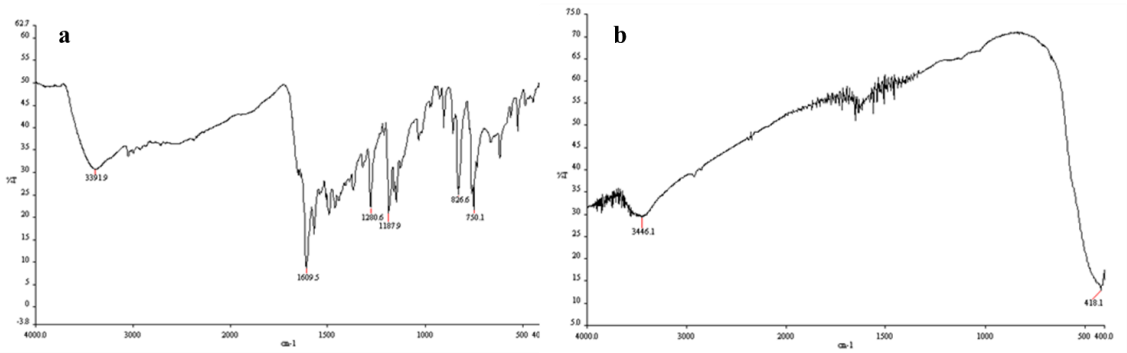

Fig. 1. FT-IR spectra of : a) nickel precursor and b) as prepared NiO nanoparticles

3.2. XRD pattern

XRD pattern of the as prepared NiO nanoparticles is shown in Figure 2, it indicates the formation of cubic nickel oxide particles (JCPDS card No. 78-0643) [25]. Comparing the XRD pattern to the previous reports [9-14], it can be concluded that NiO nanoparticles have a higher crystallinity. Also, no characteristic peaks of impurity were observed. The diffraction peaks at 37.27, 43.27, 62.84, 75.42 and 79.36° of 2θ correspond to the (111), (200), (220), (311) and (222) planes, respectively [9-14].

3.3. SEM and TEM images

After characterization of the as prepared NiO nanoparticles by FT-IR and XRD, for the determination of the morphology and structure of NiO, SEM and TEM measurements were carried out (Fig. 3). Morphology evolution of prepared NiO nanoparticles showed spherical shapes, aggregated with an average size of 78 nm. Furthermore, the geometrical structures of NiO were elucidated by TEM which clearly shows that the NiO are nearly plate with an average size between 10-40 nm.

4. Conclusions

In summary, pure and nearly uniform and plate-like NiO nanoparticles with an average size between 10-40 nm were successfully prepared by thermal decomposition of nickel (II) Schiff base precursor at 450°C. This method is simple, low-cost, environmentally friendly and suitable for the preparation of NiO nanoparticles. This method can also be used to synthesize nanoparticles of other metal oxides.

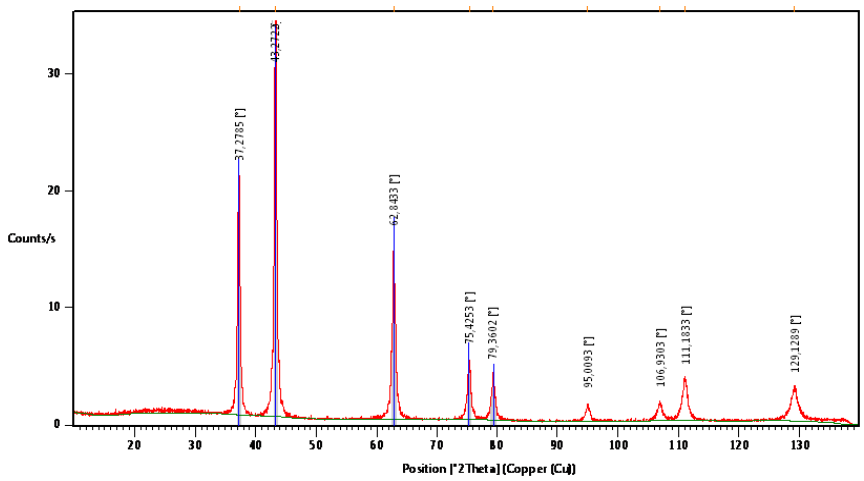

Fig. 2. XRD pattern of as prepared NiO nanoparticles

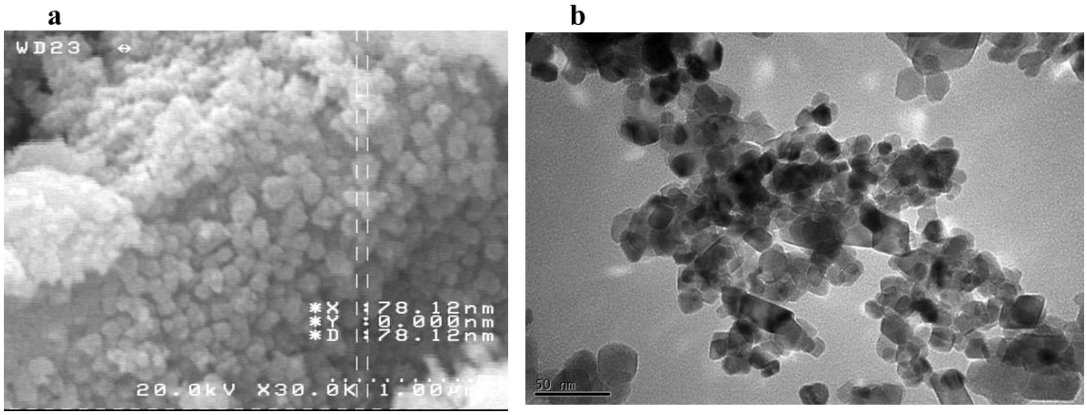

Fig. 3. Micrographs of as prepared NiO nanoparticles: a) SEM and b) TEM

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