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ORIGINAL ARTICLE

Synthesis and characterizations of NiO nanoparticles via solid-state thermal decomposition of nickel(II) Schiff base complexes

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Abstract To raise the need of new precursors in the synthesis of NiO nanoparticles, mononuclear nickel(II) Schiff base complexes, viz. Ni(salbn) and Ni(Me_2-salpn), were employed as precursor in solid-state thermal decomposition. Structure, purity and morphology of these nanoparticles have been examined by Fourier transform infrared spectroscopy, X-ray powder diffraction, scanning electron microscopy and transmission electron microscopy (TEM). TEM analysis reveals that the synthesized nanoparticles have cubic particles with an average diameter of around 5–15 nm. This method is simple, less costly, and fast and safe for production of NiO nanoparticles in industrial applications.

Keywords Nickel oxide · Schiff base precursors · PXRD · SEM · TEM

Background

Recently, nickel(II) Schiff base complexes have been widely investigated not only for their interesting structures and properties [1, 2], but also for their use as precursor for preparation of nickel oxide nanoparticles [3]. Then, considerable interest has been grown on the preparation and characterization of NiO nanoparticles via thermal decomposition of complexes for their unique applications and properties [4, 5]. Among the transition metal oxides, nickel oxide has attracted much attention due to its properties and applications [6, 7]. Presently several methods, viz. hydrothermal, thermal decomposition, sol–gel, solvothermal and sonochemical are available to prepare NiO nanoparticles [8–10]. However, thermal decomposition method is a better choice as it makes control process conditions, particle size, particle crystal structure and purity possible [3–5]. Salavati-Niasari et al. have reported the synthesis of NiO nanoparticles [3] using four coordinated nickel(II) Schiff base complex Ni(salen) while Farhadi group have synthesized NiO nanoparticles by solid-state thermal decomposition of octahedral nickel(II) complexes, viz. $[Ni(en)_3](NO_3)_2$ [4] and $[Ni(NH_3)_6](NO_3)_2$ [5].

Recently, our group has synthesized nickel oxides nanoparticles via thermal decomposition method of nickel Schiff base complexes [11–13]. Herein, we have reported the synthesis of NiO nanoparticles by solid-state thermal decomposition of Ni(II) Schiff base complexes, Ni(salbn) (**1**) and Ni(Me_2-salpn) (**2**) (Scheme 1).

Experimental

Materials and characterization

All reagents and solvents for synthesis and analysis are commercially available and used as received. Elemental analyses are carried out using a Heraeus CHN-O-Rapid analyzer. X-ray powder diffraction (PXRD) pattern of the nanoparticles is recorded on a Panalytical Empyran with Cu–K_α radiation in the range $2\theta = 0°$–$140°$. Fourier transform infrared spectroscopy (FTIR) spectra are recorded as a KBr disk on a Perkin–Elmer FTIR spectrophotometer. Scanning electron microscopy (SEM) images are

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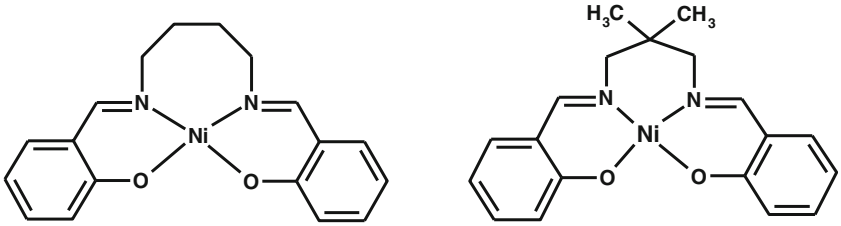
Scheme 1 Chemical structures of Ni(salbn) (**1**) and Ni(Me_2-salpn) (**2**)



Ni(salbn) (1) **Ni(Me_2-salpn) (2)**

collected on Philips XL-30ESEM. Transmission electron microscopy (TEM) images are recorded on a JEOL JEM 1,400 instrument with an accelerating voltage of 120 kV.

Preparation of Ni(salbn) (**1**) and Ni(Me_2-salpn) (**2**) complexes

To obtain a dark red precipitates of complexes, a methanol solution (20 mL) of salbn or Me_2-salpn (2 mmol) was added to methanol solution (20 mL) of $Ni(NO_3)_2{\cdot}6H_2O$ (2 mmol). The resulting solution was stirred for 2 h. The product was removed by filtration, washed with cooled ethanol and dried at room temperature for several days. Anal. calcd. for $C_{18}H_{18}NiN_2O_2$ (**1**): C, 61.24; H, 5.13 and N, 7.93 %; found C, 61.36; H, 5.23 and N, 7.86 %. FTIR (cm^{-1}): 1,624 (C=N). Anal. calcd. for $C_{19}H_{20}NiN_2O_2$ (**2**): C, 62.17; H, 5.49 and N, 7.63 %; found C, 62.26; H, 5.53 and N, 7.76 %. FTIR (cm^{-1}): 1,610 (C=N).

Preparation of NiO nanoparticles

New precursors **1** and **2** are loaded on to a platinum crucible and placed in an oven to be heated at a rate of 10 °C/min in air. Nanoparticles of nickel oxide are synthesized at 450 °C after 3.5 h. To remove any impurities, the final products are washed with ethanol and dried at room temperature for 3 days.

Results and discussion

FTIR spectra

The FTIR spectra of the precursors **1** and **2** are shown in Fig. 1. The characteristic peaks at 1,624 in **1** and 1,610 cm^{-1} in **2** are attributed to the υ(C=N) stretchings [3–5] which disappeared in Fig. 2 with the appearance of new strong band at 418 cm^{-1} in nanoparticles prepared from **1** and at 420 cm^{-1} in nanoparticles prepared from **2**, indicating the spinel structure of Ni–O [3–5]. The broad band at around 3,500 cm^{-1} in the spectrum of NiO nanoparticles is attributed to the adsorbed water on the external surface of the samples. Also, the H-OH bending vibration appears at about 1,640 cm^{-1} [3–5].

XRD patterns

Figure 3 shows the XRD patterns ($0° < 2\theta < 130°$) of the nickel oxide nanoparticles prepared via solid-state thermal decomposition from nickel(II) Schiff base complexes **1** and **2**. The powders showed the crystalline pattern, and according to standard nickel oxide pattern (JCPDS: 75-0197) [3–5], all diffraction peaks can be well indexed as face-centered cubic phase at about $2\theta = 37°$, 43°, 62°, 75° and 79°, which can be perfectly related to 111, 200, 220, 311 and 222 crystal planes, respectively. No obvious peaks of impurities were seen in the XRD pattern of NiO obtained by thermal decomposition. Moreover, the observed peaks are sharper and higher in intensity which confirmed the well crystallization of the prepared NiO nanoparticles. These results confirm that at 450 °C the complexes were decomposed completely to nickel oxide.

SEM and TEM images

The morphology and microstructure of the NiO nanoparticles have been investigated using SEM and TEM techniques. Figures 4 and 5 are the respective SEM and TEM images of the nanoparticles obtained from **1** and **2** which indicate their particle size around 10–15 nm. The particles are both spherical and cubic. The choice of nickel precursor and method is the main step in the synthesis of nickel oxide nanoparticles (Table 1). Although many different precursors and methods have been used for preparation of nickel oxide [3–5], this report is the scare report on the synthesis of NiO nanoparticles from nickel(II) Schiff base complexes [5–7].

Conclusions

Pure NiO nanoparticles having average size of 10–15 nm have been successfully prepared by solid-state thermal decomposition (at 450 °C for 3 h) of Ni(II) Schiff base

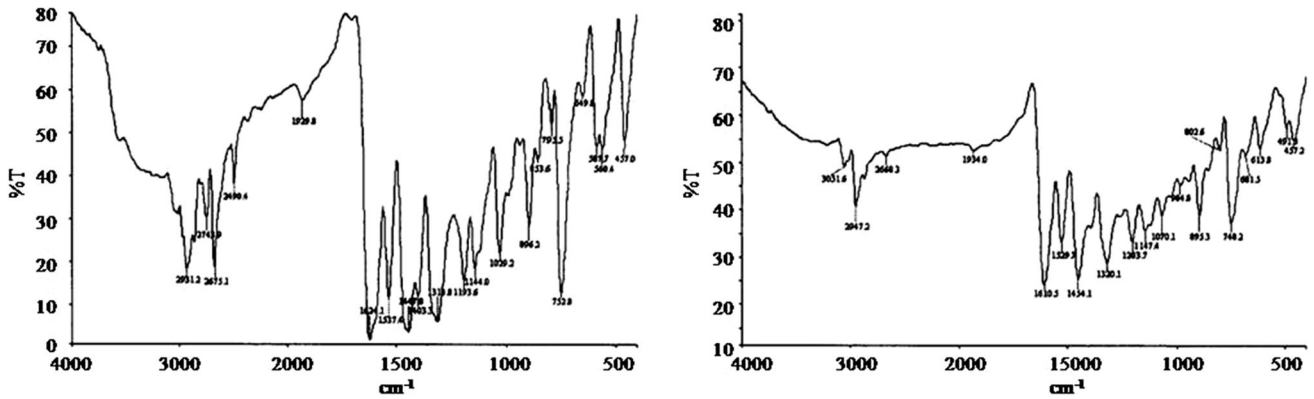


Fig. 1 FTIR spectra of Ni(II) complexes **1** (*left*) and **2** (*right*)

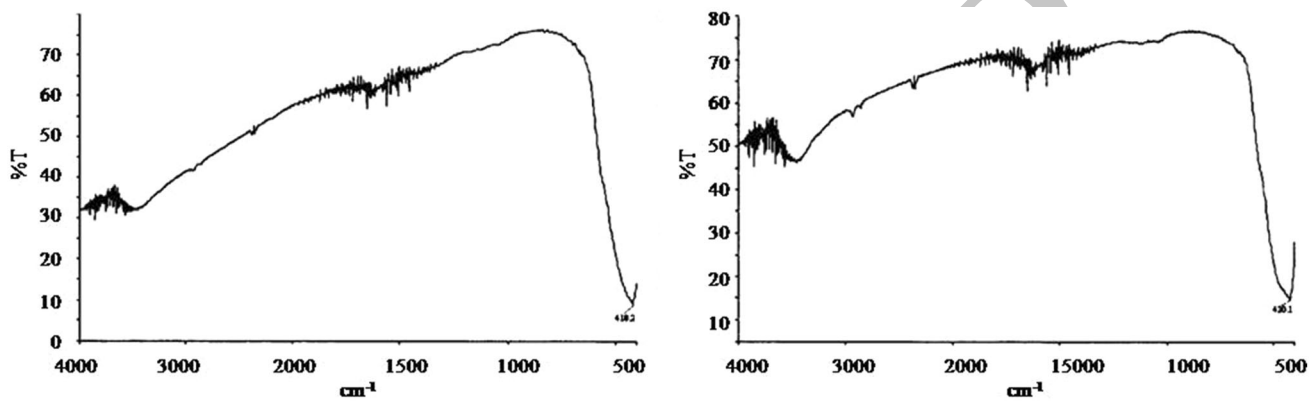


Fig. 2 FTIR spectra of NiO nanoparticles derived from **1** (*left*) and **2** (*right*)

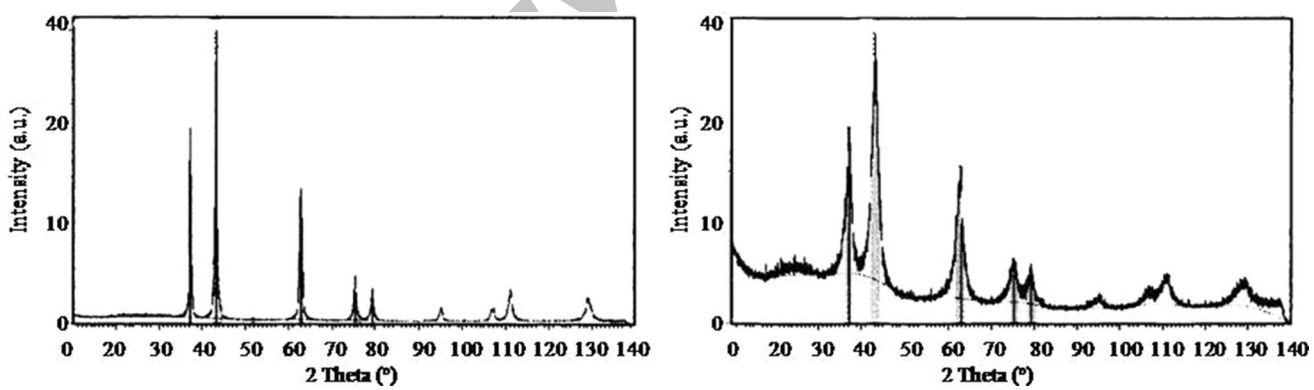


Fig. 3 XRD patterns of NiO nanoparticles prepared from **1** (*left*) and **2** (*right*)

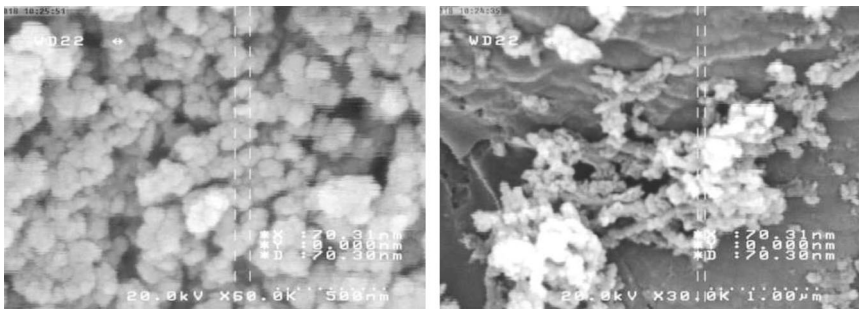


Fig. 4 SEM images of NiO nanoparticles derived from **1** (*left*) and **2** (*right*)

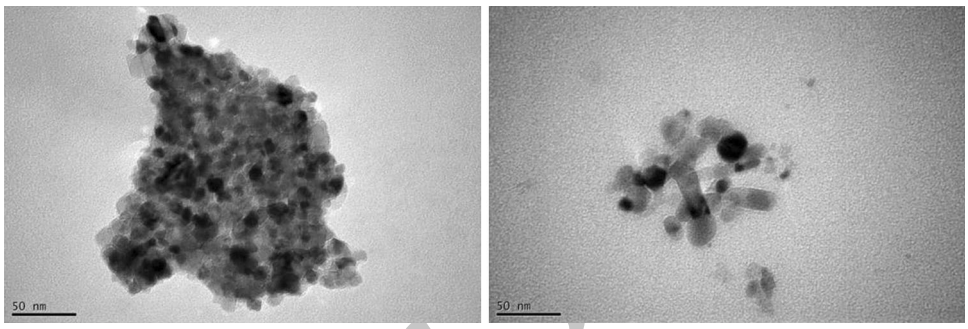


Fig. 5 TEM images of NiO nanoparticles derived from **1** (*left*) and **2** (*right*)

Table 1 Comparison of particle size of NiO nanoparticles by various methods and precursors

Precursor	Method	Particle size (nm)	References
$Ni(Phen)_2$	Thermal decomposition	56	[14]
$Ni(NO_3)_2{\cdot}6H_2O$	Sol–gel	10	[15]
$[Ni(C_7H_5O_3)(H_2O)_4]$	Thermal decomposition	5–15	[16]
$NiSO_4$	Ultrasonic	1–3	[17]
$Ni(octa)_2$	Thermal decomposition	25	[18]
Ni(salen)	Thermal decomposition	15–20	[3]
$Ni(Brsalph)(NO_3)$	Thermal decomposition	55	[12]
Ni(salbn) (**1**)	Thermal decomposition	5–15	This work
$Ni(Me_2\text{-salpn})$ (**2**)	Thermal decomposition	5–15	This work

complexes, viz. Ni(salbn) (**1**) and $Ni(Me_2\text{-salpn})$ (**2**). This facile, inexpensive and nontoxic method may be useful for the preparation of other transition metal oxide nanoparticles.

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