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BINUCLEAR NICKEL(II) COMPLEX AS NEW PRECURSOR FOR SYNTHESIS OF NIO NANOPARTICLES BY SOLID-STATE THERMAL DECOMPOSITION METHOD

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ABSTRACT

This study focuses on the synthesis of Ni (II) Schiff base complex obtained from salicylaldehyde and thiosemicarbazide. The complex was synthesized by template method and characterized by elemental analysis (CHNS), FT-IR, UV–Visible spectroscopy, conductivity measurement and magnetic moment. The spectroscopic studies suggested the square-planar structure for Ni (II) complex, respectively. Then the complex was used as precursor for preparation of nickel oxide nanoparticles via solid-state thermal decomposition without using a catalyst, toxic solvent, template or surfactant and complicated equipment, which makes it efficient, one-step, simple and environment-friendly. The chemical structure of the metal oxides is studied by FT-IR, XRD and SEM.

KEY WORDS

Salicylidenic Schiff base complex, Thermal decomposition, FT-IR, XRD, SEM.

INTRODCTION:

Life threatening infections and diseases spreading all over the world, day by day, demand development of new antimicrobial agents, that are highly efficient with lesser side effects and equally cost effective.[1] Metal complexes are preferred to simple ligands due to an increased activity and several sulphur containing metal chelates have been reported with significant antibacterial and antifungal activities.[2] A large majority of Thiosemicarbazones have been extensively studied, because of their antibacterial, antifungal, antitumor, antiviral and anticancer activities.[3] Transition metal complexes of thiosemicarbazone have attracted chemists due to the presence of hard nitrogen and soft sulphur donor atoms which permits coordination with a metal ion in different binding mode yielding stable and intensely coloured complexes.[4]

Recently, nickel (II) Schiff base complexes have been widely investigated not only for their interesting structures and properties [5] but also for their use as precursor for preparation of nickel oxide nanoparticles. 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 [6] Among the transition metal oxides, nickel oxide has attracted much attention due to its properties and applications. [7] Presently several methods, viz. hydrothermal, thermal decomposition, sol–gel, solvothermal and sonochemical are available to prepare NiO nanoparticles. However, thermal decomposition method is a better choice as it makes control process conditions, particle size, particle crystal structure and purity possible. [8-10] In this work, we wish to report the synthesis and characterization of mononuclear Schiff base nickel (II) complex.

Subsequently, direct thermal decomposition process of the desired complex precursor is one of the most important and straightforward strategies to access structurally elaborated and pure NiO nanoparticles with regular spherical particle.

MATERIALS AND METHODS:

Materials and Reagents

All chemicals were of analytical grade and purchased from Merck and Sigma Aldrich. Commercial solvents were distilled and then used for the preparation of Schiff base copper (II) complex

Instruments:

Elemental analysis was performed using a Perkin-Elmer elemental analyzer. Molar conductivity of the metal complex was determined by using DMF as a solvent in Equiptronics digital conductivity meter at room temperature. FT-IR spectra of Schiff base copper (II) complex and its copper oxide nanoparticles were obtained on a Schimadzu IR-Affinity-I spectrometer with samples prepared using KBr pellets. UV–Visible spectra were recorded using Systronics spectrophotometer operating in the range of 200–800 nm with quartz cell. X-ray powder diffraction (XRD) pattern of the complex was recorded on a Bruker AXS diffractometer D8 ADVANCE with Cu-Kα raiation ith nickel beta filter in the range 2Θ = 10º-80º. Scanning electron microscopy (SEM) images were obtained on Philips XL-30ESEM.

Synthesis of Schiff Base Nickel (II) complex:

Thiosemicarbazide (10mmol) in hot ethanol(30ml) was taken to which salicylaldehyde (10mmol) in 30 ml ethanol was added and the reaction mixture was refluxed for 2 hrs. After that potassium hydroxide (10mmol) in 20 ml ethanol was added and stirred for another 15 minutes Then 25ml ethanolic solution of Nickel chloride was finally added to the above reaction mixture and stirred for 1hour. Green coloured complex was formed which was washed several times with alcohol and water, filtered and air dried.

Synthesis of nickel oxide nanoparticles by thermal decomposition method:

Schiff Base Nickel (II) complex precursor was taken in a silica crucible and heated to 400° C in a muffle furnace at a ramping rate of 10°C min^{-1} for 3hours. The reaction heated to 400° C at a ramping rate of 10°C min^{-1}, the Green colored complex turned to black, which implied that Nickel oxide nanoparticles had been produced. Nanoparticles were cooled down to room temperature.

RESULTS AND DISCUSSION:

The synthesised Schiff base nickel (II) complex was characterised by Elemental analysis, UV-Visible, FT-IR and Molar conductance, Magnetic susceptibility measurement and the results are given below.

Elemental analysis:

The elemental analysis data of Schiff base nickel (II) complex is given in Table 1. The calculated value was in good agreement with the theoretical value

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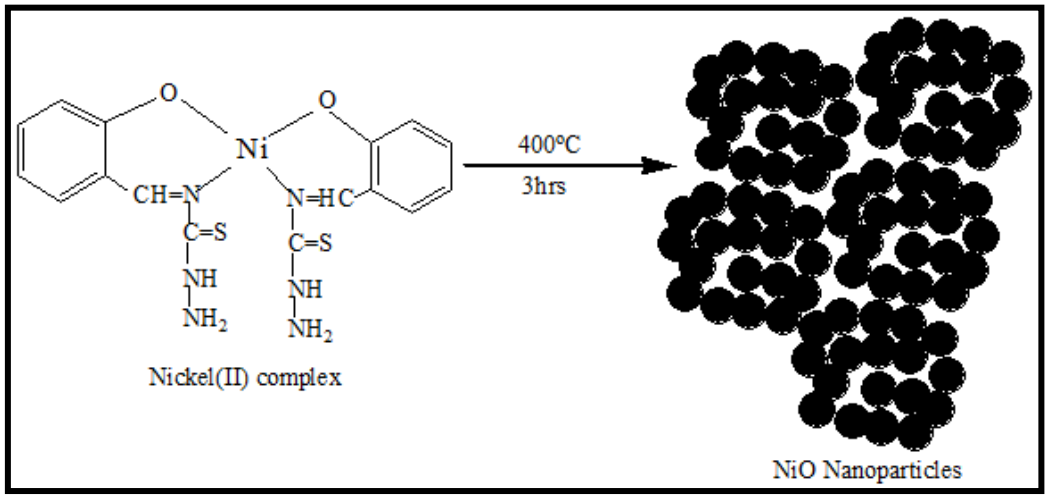

Table 1: Analytical and physical data of Schiff base nickel (II) complex

Compound	Empirical Formula	μ_{eff} BM	Molar Conductance (mho cm^2 mol^{-1})	Elemental Analysis Found (Cal.) (%)		
				C	H	N
Complex-I	$C_{16}H_{16}O_2N_6S_2Ni$	1.92	55	43.10 (43.05)	3.52 (3.59)	18.89 (18.83)

Molar conductivity and magnetic susceptibility measurement:

The molar conductance values measured in DMF solution ($1x10^{-3}$ mol dm^{-3}) and the value is 55 ohm^{-1} cm^2 mol^{-1} suggesting that the complex is non-electrolyte.[11] The room temperature magnetic moment (μ_{eff}) value of the nickel(II) complex is observed in 1.92 B.M., which corresponds to a single unpaired electron and may be concluded that the copper(II) complex has square planar geometry.

Electronic Spectra:

The electronic absorption spectra of Schiff base nickel (II) complex (10^{-3} M) was recorded in DMF at room temperature and the data is represented in the Table 2. The band appeared around 293 nm and 360 nm corresponds to π→π* transition of aromatic chromophore as well as the imine moiety and the band at 435 nm is due to n→π* transition respectively. [12] The copper (II) complex exhibited a broad band at 680 nm due to d-d transition.

Table 2: UV-Visible spectral data of Schiff base nickel (II) complex

Compound	π-π* (nm) (benzene)	π-π* (nm) (-HC=N)	n-π* (nm)	d-d (nm)
Complex-I	293	360	435	680

FT-IR Spectra:

The FT-IR spectrum provides valuable information regarding the nature of the functional group attached with the metal ion in the synthesized Schiff base nickel (II) complex. The synthesized Schiff base nickel (II) complex was characterized mainly using the C=N (azomethine), M-O, M-N peaks. The assignments of important infrared spectral data are listed in Table 3. The peak observed in the range 1604 cm^{-1} was characteristic of azomethine (HC=N-) group. [13] The formation of M-N and M-O linkage was confirmed the appearance of peaks around 580 and 460 cm^{-1} respectively. The characteristic peak at 1604 cm^{-1} which is attributed to the (C=N) stretching is disappeared in FT-IR spectra of nickel oxide nanoparticles whereas a new strong peak was appeared at 430 cm^{-1} in nanoparticles prepared from nickel (II) complex indicating the spinel structure of Ni–O.

Table 3: FT-IR spectral data of Schiff base nickel (II) complex

Compound	υ(-OH)	υ_{aro}(C-H)	υ(CH=N)	υ(Cu-N)	υ(Cu-O)
Complex-I	3169	2960	1604	580	460

XRD Analysis:

Powder X-ray Diffraction (XRD) is one of the primary techniques used by mineralogists and solid state chemists to examine the physico-chemical make-up of unknown materials. X-ray diffraction is one of the most important characterization tools used in solid state chemistry and materials science. XRD is an easy tool to determine the size and the shape of the unit cell for any compound. Powder Diffraction Methods is useful for Qualitative analysis (Phase Identification), Quantitative analysis (Lattice parameter determination & Phase fraction analysis) etc. Diffraction pattern gives information on translational symmetry - size and shape of the unit cell from Peak Positions and information on electron density inside the unit cell, namely where the atoms are located from Peak Intensities. [14]

Fig.1 shows the XRD pattern of sample synthesized by thermal decomposition method with dry temperature *Td* of 400°C for 3 hrs. The spectra are almost equal to the typical XRD spectra of NiO nanoparticles reported from other experiments. In all our XRD patterns nine peaks are observed around 2Θ = 33.14, 34.46, 38.6,

48.66, 57.54, 62.52, 68.95, 70.23, 75.24 which correspond to (100), (002), (101), (102), (110), (103), (200), (112) and (201), respectively. For this sample, all observed peaks can be indexed as the hexagonal wurzite structure of NiO with having space group P63*mc*. The crystalline size of the NiO nanoparticles based on X-ray peak broadening were determined using the Debye-Scherrer equation d (Å) = 0.9 λ / β cosθ. The calculated values are about 58 nm for NiO nanoparticles prepared from Schiff base nickel (II) complex.

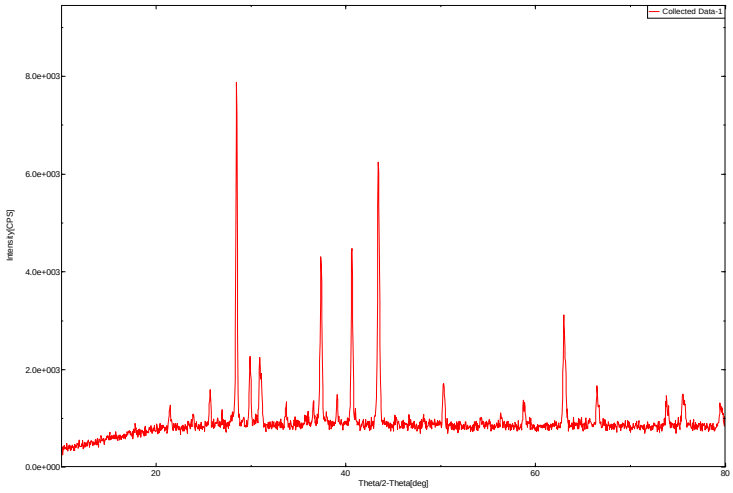

Figure 1: XRD Pattern of NiO nanoparticles

SEM image:

The Morphology of synthesized NiO Nanoparticles was investigated by scanning electron microscopy (SEM). Fig 2 shows the SEM image of NiO nanoparticles synthesized by thermal deomposition method and found to be flower-like shape. As seen from the SEM image, NiO nanoparticles have lower particle size. Particles were found to be less than100 nm but were in agglomerated form. [15]

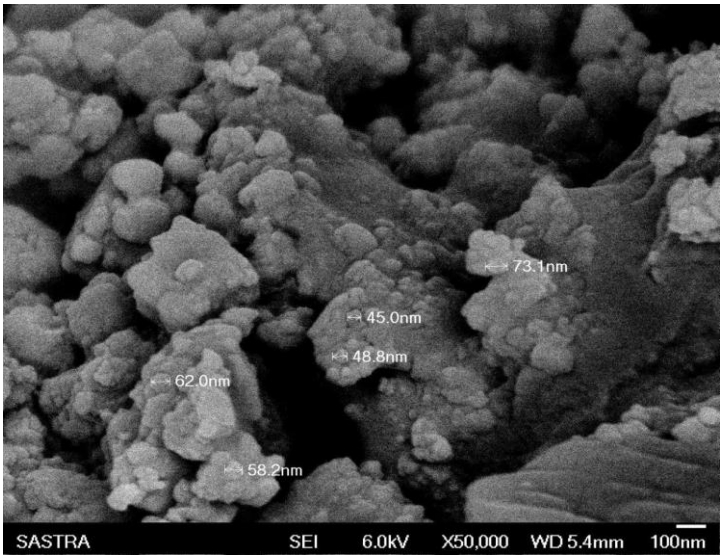

Figure 2: SEM image of NiO nanoparticles

CONCLUSION:

In this paper, Schiff base complex of Ni^{2+} from salicylaldehyde and thiosemicarbazide was synthesized and characterized. Then pure NiO nanoparticles with 56 nm in size have been successfully synthesized through the decomposition of Schiff base complex using muffle furnace. The solid-state thermal decomposition method is an easy, safe and suitable for high purity production

for the preparation of nanoparticles. This method also has potential advantages, including operational simplicity, no need for solvent, low energy consumption and no special equipment required.

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