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Sono chemical syntheses of a new nano-sized Zn (II) Schiff base complex as a precursor for the preparation of Zn (II) oxide nanoparticles by calcination process

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Abstract : In the present work, novel nano-sized ZnO powder was prepared via nano-sized Zn (II) complex as a precursor compound by calcination process. Nano-sized of a new Zn(II) complex, [Zn((E)-4-((2-hydroxyl)imino)pentan-2-one)(OH_2)] were synthesized by a sonochemical method. The new nano-structure was characterized by scanning electron microscopy, elemental analyses and IR spectroscopy. After calcination of this nano-sized Zn(II) complex at 400 °C, pure phase nano-sized Zn(II) oxide has been produced. The produced nano sized ZnO was characterized by X-ray powder diffraction (XRD) and scanning electron microscopy (SEM).

Keywords : Sono chemical; Schiff base complex; Nanoparticle; Zn(II) oxide; Calcination.

INTRODUCTON

Imines compounds as Schiff bases, named after Hugo Schiff[1], are formed when any primary amine reacts with an aldehyde or a ketone under specific conditions. Structurally, is a nitrogen analogue of an aldehyde or ketone in which the carbonyl group (-C=O) has been replaced by an imine or azomethine group (-HC=N-).

Many complexes of Schiff-base ligands with metal ions have been investigated as models for active sites of enzymes[2,3], including DNA-cleavage systems[4,5], and as antibacterial[6-8] and anticancer[9] drugs. They also provide useful magnetic materials[10] and have a wide range of catalytic applications, such as in polymerization[3], olefin oxidation[4] and Suzuki-Miyaura coupling[5]. In particular, complexes of metal ions with tetrahedral (e.g. Ag(I)) or octahedral (e.g. Cu(II), Co(II), Fe(II) and Zn(II)) coordination preferences have been found to display important physicochemical properties[6,9,11,12] as well as biological activity[7-10].

Nano-structured materials with uniform morphology have stimulated great interest due to their importance in basic scientific research and potential technology applications[13,14]. The ability to control the shape and size of these nanomaterials is one of the most challenging issues in chemistry and materials science.

Zinc oxide is a chemical compound found naturally in the mineral called zincite and has attracted much attention in recent times due to its low cost and because it can be obtained by simple techniques[15]. Nanocrystalline ZnO powders- due to their average particle size (below 100 nm) may show different behaviors resulting from a higher surface energy due to the large surface

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area and wider gap between the valence band and conduction band, effects characteristic of sizes close to the atoms. These phenomena may increase the potential use of the material, including optical, chemical, and electromagnetic, among other properties. Therefore, because of its exceptional physical and chemical characteristics[16], the nano zinc oxide (ZnO) ia an important raw material for many applications as the design of various, gas sensors, luminescent oxide, rubbers, paints, ceramics, and others[17,18].

This work deals with the synthesis and characterization of a Schiff base (E)-4-((2-hydroxyethyl)imino)-pentane -2-one [HIPO] obtained by the condensation of 2, 4-pentanedione (acetylacetone) with 2-aminoethanol and its nano-sized Zn(II) complex. The coordination behavior of Schiff bases towards transition metal ion was investigated via the FT-IR study. The morphology of its structure was study by X-ray powder diffraction (XRD) and scanning electron microscopy (SEM). The pure nano-sized ZnO powder has been produced after calcination of this nano- sized Zn(II) complex at 400 °C. The produced nano sized ZnO was characterized by X-ray powder diffraction (XRD) and scanning electron microscopy (SEM).

EXPERIMENTAL PROCEDURE

Synthesis of (E)-4-((2-hydroxyethyl)imino)pentane -2-one [HIPO]

HIPO compound as a tridentate Schiff base ligand compound (Scheme 1) was prepared by the procedure reported in the literature[19].

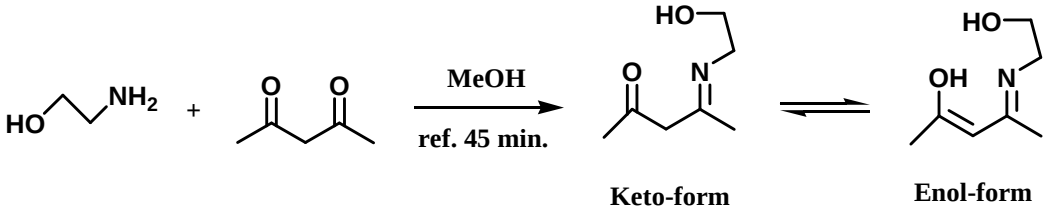
The reaction mixture containing acetylacetone (0.01 mol, 1.0012 g) and 2-aminoethanol (0.01 mol, 0.6108 g) in 10 ml methanol was refluxed at 65 °C for about 45 min. The reaction mixture after cooling at room temperature produced white crystals, which were filtered and recrystallized in methanol. Yield, 77%; m1344(m), p. 74-76 °C. FT-IR (KBr) ν_{max}: 3415 (br, m), 2865(w), 2997(w), 2877(w), 1620(s), 1433(m), 1367(m), 1313(m), 1244(m), 1193(w), 1114(w), 1075(w), 1050(w), 1023(w) cm^{-1}.

Synyhesis of nano-sized Zn(II) Schiff base complex

To prepare nano-sized Zn(II) Schiff base complex, 0.1097 g (0.50 mmol) Zn(acetate), 2H2O were disoled in 10 ml methanol. This mixture was added under ultrasound irradiation with drop by drop about 45 min. into methanolic solution of 0.0706 g (0.50 mmol) HIPO Schiff base ligand. Nano-sized Zn(II) Schiff base complex was obtained [Scheme 2]. FT-IR (selected bands; 631(w), 729(m), 810(m), 1033(m), 1270(m), 1469(m), 1639(s) cm^{-1}): The whit precipitates was then filtered and rinsed two times with warm methanol and dried at 60 °C for 2h Yield, 54% [Scheme]. The crystalline structure of this complex was characterized by X-ray diffraction (XRD, PHILIPS, X,pert-MPD system, λ=1.54 Å), FT-IR spectra were recorded in the 400-4000 cm^{-1} range with a TENSOR 2 detector and KBr pellet technique. The morphology and average particle size of nono-sized Zn(II) complex were further investigated by scanning electron microscopy (SEM, PHILIPS, XL 300).

Preparation of Zn(II) oxide nanoparticles by direct calcination

The nano-sized ZnO were prepared by heating of nano-sized Zn (II) Schiff base complex in an electrical furnace (THERMOLYNE 1500) at 450 °C for about 2 h. The crystalline structure of nano-sized ZnO characterized by X-ray diffraction (XRD, PHILIPS, X·pert-MPD system, λ=1.54Å), FT-IR spectra were recorded in the 400-4000 cm^{-1} range with a TENSOR 2 detector and KBr pellet technique. The morphology and av-



HIPO

Scheme 1 : Synthesis diagram of (E)-4-((2-hydroxyethyl)imino)pentane -2-one [HIPO] Schiff base ligand

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(*E*)-4-((2-hydroxyethyl)imino)pentan-2-one $\xrightarrow[\text{MeOH}]{\text{Zn(acetate)}_2,\ 2H_2O}$ **[Zn((E)-4-((2-hydroxyl)imino)pentan-2-one)(OH2)]**

[HIPO]

Scheme 2 : Synthesis diagram of Zn (II) HIPO Schiff base complex

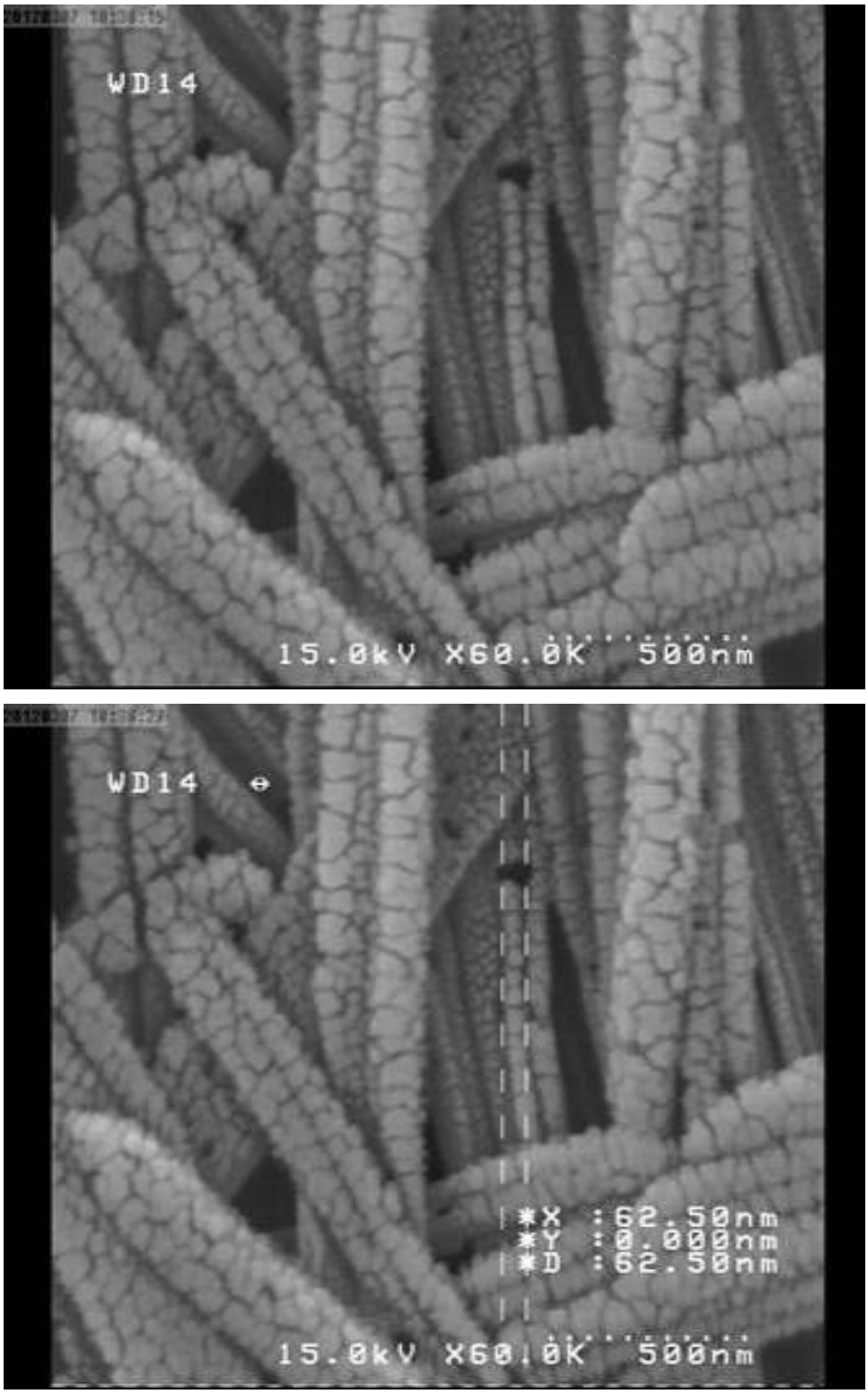

Figure 1 : SEM image of nano-sized Zn(II) Schiff base complex

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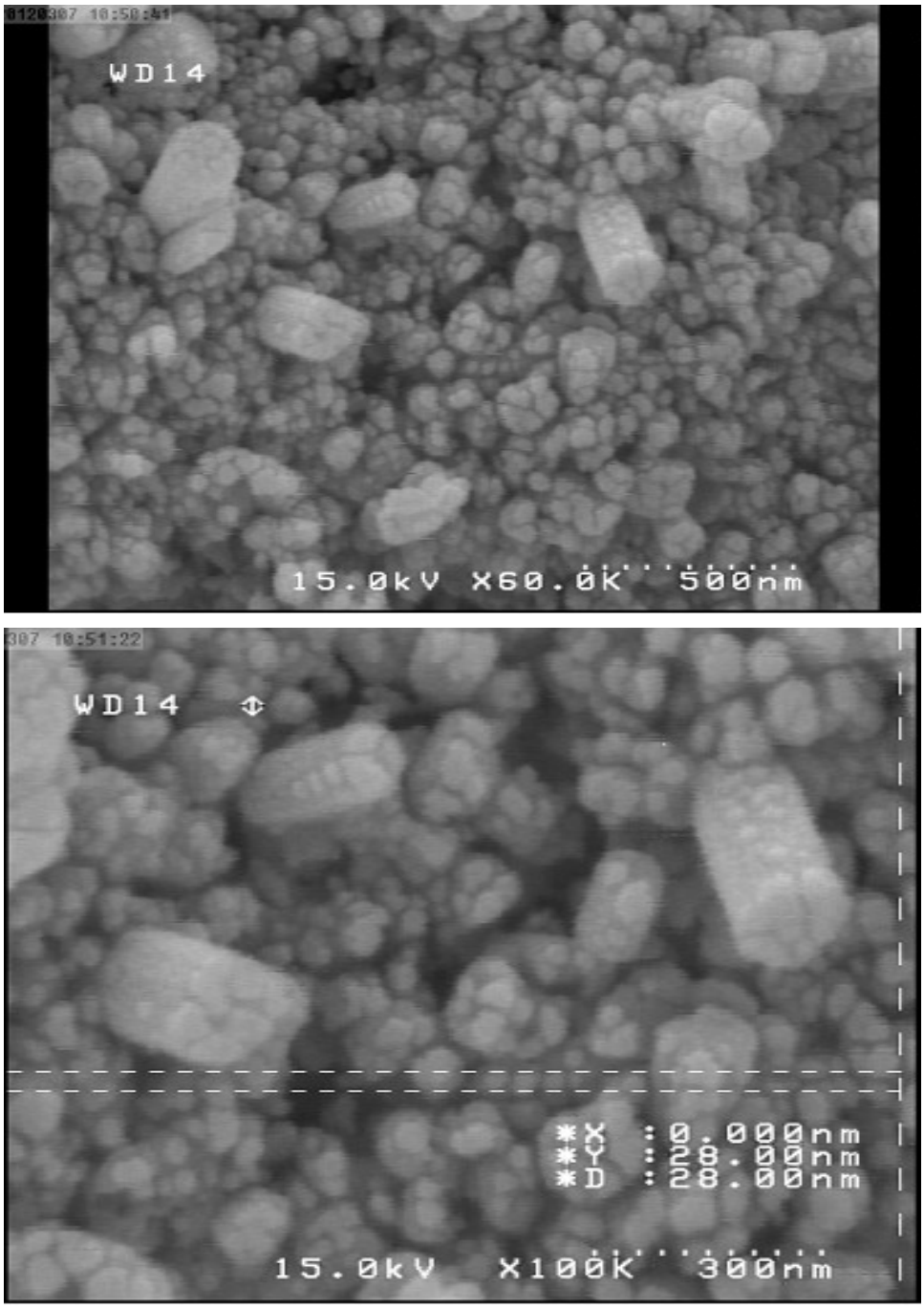

Figure 2 : SEM image of nano-sized Zn(II) oxide

erage particle size of nono-sized ZnO were further investigated by scanning electron microscopy (SEM, PHILIPS, XL 300).

RESULTS AND DISCUSSION

White pure nano-sized of Zn(II) Schiff base complex were prepared, and its structural was investigated by FT-IR spectroscopic method. IR spectra of this compound has shown some important absorption at 1639 cm^{-1} (C=N), 1270 cm^{-1} (C-O), 631, 729 and 810 cm^{-1} (m) Zn-O. The morphology and size of the nanostructure of Zn(II) complex were investigated by Scanning Electron Microscopy (Figure 1). The observed particle size is in the 62 nm.

Figure 2 shows the SEM image for nano-sized ZnO from the decomposition of nano-sized Zn(II) Schiff base complex at 450 ^{o}C by direct calcination. The observed

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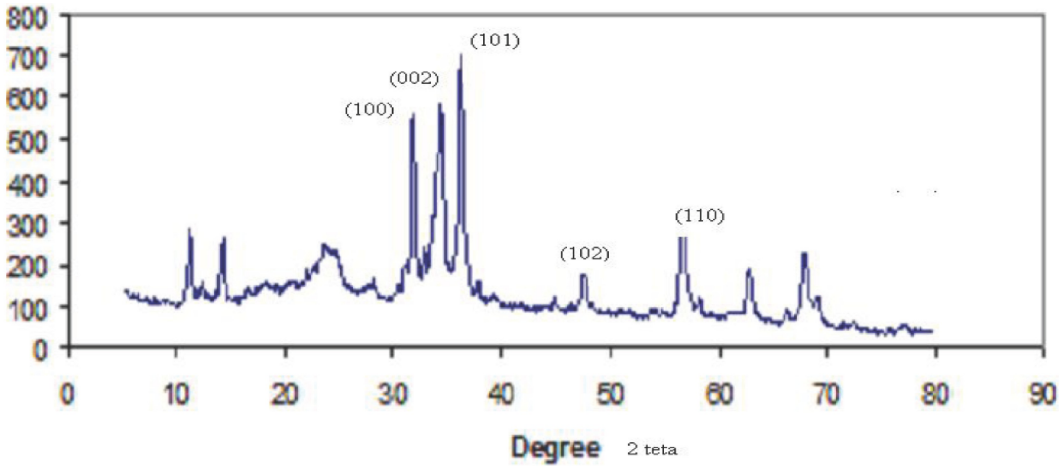

Figure 3 : The XRD pattern of nano-sized ZnO by calcinaton of Zn(II) Schiff base complex

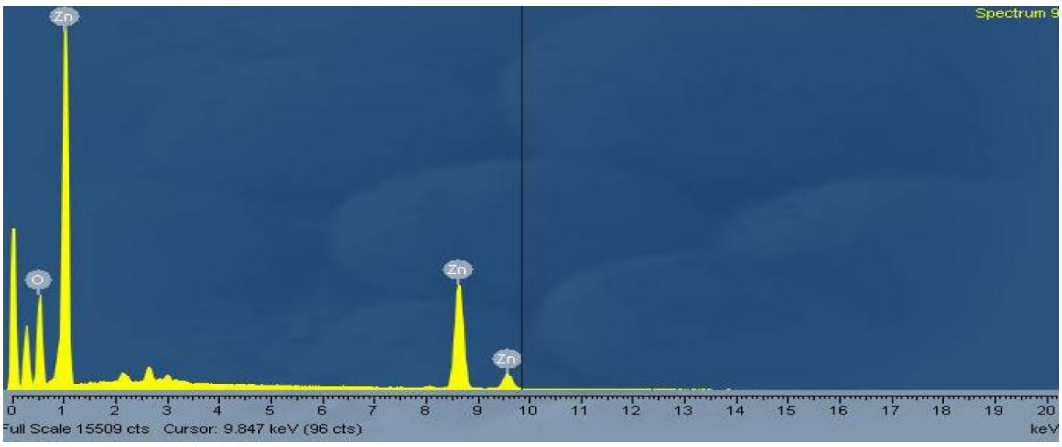

Figure 4 : EDAX pattern of nano-sized ZnO

particle size is in the 28 nm.

Figure 3 shows the XRD pattern of the colorless nano-sized ZnO sample after calcination nano-sized Zn(II) complex for 2 hours at 450 °C. The XRD pattern of the standard ZnO with lattice parameters (Sys: Hexagonal, S.G. $p6_3mc$, a=3.2498 Å, c= 5.2066 Å and z=2) which are the same as the reported values (JCPDS card No. 36-1451). Sharp diffraction peaks shown in Figure 3 indicate good and pure phase crystallinity of ZnO nanoparticles.

The energy-dispersive_X-ray_spectroscopy (EDAX) pattern of nano-sized ZnO shows the presence of zinc and oxygen as the elementary compound (Figure 4).

CONCLUSION

In this study it was shown that ZnO nano-particles can be produced by direct calcination method of a zn(II0 Schiff base complex as the precursor, at 450 C for 2 h. XRD, EDAX analysis confirmed the consist of ZnO and the mean particles size of ZnO nano-particles determined by SEM analysis was about 96 ±5 nm in diameter and length, respectively. Figure 3 shows the XRD pattern of ZnO nano-crystals. The result shows that all the diffraction peaks are indexed to ZnO with standard structure (JCPDS card No. 36-1451).

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