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Synthesis of Nanoparticles by using Schiff Base Cu (II) and Zn (II) Complexes as a Precursor and its Antibacterial Studies

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Abstract: ***Metal Schiff base complexes have been widely investigated not only for their interesting structures and properties but also for their use as precursor for preparation of metal oxide nanoparticles. Considerable interest has been grown on the preparation and characterization of nanoparticles via thermal decomposition of complexes for their unique applications and properties. Among the metal oxides, transition metal oxides have attracted much attention due to its properties and applications. Schiff base ligands of different amines and aldehydes were prepared by condensation method. Nanoparticles were prepared using these ligands metal complexes as precursor. Structure of these nanoparticles have been examined by X-ray powder diffraction reveals the size of nanoparticles. Antibacterial potential of the prepared nanoparticles were carried out by using gram negative E.Coli bacteria and shows good antibacterial potential.***

Keywords: ZnO nanoparticles, CuO nanoparticles, Antibacterial study

1. Introduction

Nanotechnology is an interdisciplinary area of science which has been growing and developing rapidly across the world forming nano revolution by producing nanoproducts and nanoparticles that can have novel and size related physico-chemical properties differing significantly from larger matter. [1]. Nano particles have become important materials in modern technologies compared with their bulk analogues not only as a result of their excellent structural features but also as a result of their unusual attributes[2]. Nanostructure metal oxides have been attended in the field of nanotechnology both from a fundamental and industrial point of view. Metal oxides play a very important role in many areas of chemistry; physics and material science. The metal elements are able to form a large diversity of oxides[3-6]. Transition metal oxides and metals have been researched extensively due to their interesting catalytic, electronic and magnetic properties. Nanometer sized metal oxides and metals find wide applications in data storage devices, catalysis, drug delivery and biomedical imaging[7-9]. Presently several methods, viz. hydrothermal, thermal decomposition, sol–gel, solvothermalsonochemical etc. are available to prepare nanoparticles. However, thermal decomposition of transition metal is one of the simplest and less costly techniques for preparing nanosized transition metal oxides[10-13]. Copper oxide is an important transition metal oxide with many practical applications, such as it is the basis of several high-Tc superconductors and materials with giant magnetoresistance, and is also used as catalysts, pigment, p-type semiconductor, gas sensors, solar cells, magnetic storage media and cathode materials [14, 15]. Zinc oxide is also a well-known semiconductor material with a wide band gap (3.37 eV) and a large exciton binding energy (60 meV) at room temperature. ZnO nanoparticles have been extensively studied over the past decades because of their fascinating electrical,mechanical,optical, and piezoelectric properties. ZnO nanoparticles have a wide range of applications such as gas sensors, dye-sensitized solar cells, ultra violet photodetectors, UV lasers, photocatalysts, piezoelectric transducers, and for biomedical applications [16-18]. Because of the practical reasons mentioned above, the synthesis of nanostructured CuO has also attracted considerable attention. In this paper we described the synthesis and characterization of CuO and ZnO nanoparticles by solid state thermal decomposition of Cu (VATSC),Cu(SALTSC),Zn(VATSC),Zn(SALTSC).

2. Experimental Methods[19, 20]

Preparation of Vaniline Thiosemicarbazone(VATSC)
A solution of thiosemicarbazide (0.01 mole) in ethanol was added to an ethanolic solution of Vaniline (0.01 mole). The mixture was refluxed for about 2 hours. The precipitate was then filtered, washed with ethanol and dried for 6 hours.

Preparation of Salicylaldene Thiosemicarbazone (SALTSC)
0.01 mole of salicylaldehyde was dissolved in ethanol and taken in a RB flask. To this ethanolic solution of 0.01 mole of thiosemicarbazide was added, mixed well and refluxed for two hours. Adjusted the PH by adding sodium acetate. Heated for two more hrs and allowed to cool for 4 hrs. The precipitate was then filtered, washed with ethanol and dried for 6 hours.

Synthesis of copper and zinc complexes
To an ethanolic solution of the Schiff base, an ethanolic solution of the metal Nitrate[Copper Nitrate, Zinc Nitrate respectively] was added in a molar ratio (1:1).The mixtures were refluxed for about 1hour.The resulting precipitate was collected by filtration, washed with ethanol and dried.

Preparation of ZnO and CuO nanoparticles
To prepare CuO, ZnO nanoparticles the prepared Schiff base complexes were transferred in to a porcelain crucible and placed in a muffle furnace, heated to700^{0}C for 3 hrs. To remove any impurities, the final products are washed with ethanol and dried at room temperature for 3 days.

Antibacterial studies

Disc diffusion method was carried [22-23] out by using gram negative bacteria- *E.Coli* to evaluate the antibacterial potential of the sample. Bacterial culture of *Escherchia-coli (E. Coli)* was swabbed on freshly prepared nutrient agar which was poured on sterile petridishes. After solidification, the prepared polymer samples at different concentrations (50, 75μg/mL) were loaded in discs and the plates were incubated at 37^0C for 24 hours. After incubation, the diameter of inhibitory zones formed around each well/disc was measured in mm and compared with positive control (Tetracycline).

3. Results and Discussion

The infrared spectra of VATSC and SALTSC exhibit a sharp band at 1610cm^{-1} and 1635cm^{-1} respectively due to azomethine linkage. In all the complexes this band appears at a frequency lower than that of the free ligand. This clearly indicates the involvement of nitrogen atom in coordination due to a reduction in the electron density in the azomethine linkage. Ketoenoltautomerism is possible for the thiosemicarbazone and the ligand can exist as keto or enol or mixture of both. The SH stretching vibrations are absent in the expected region 2650-2800cm-1 which indicates that the free ligand exist in the keto form in the solid state. However during the complex formation it might exist in the enol form. Thus in all the complexes studies this is clear indication for the participation of sulphur atom in co-ordination. The free hydroxyl group in a compound has stretching mode at 3700-3400^{cm-1}. The O-H band is weakened, if the group is hydrogen bonded, and in such cases the band is broadened with a shift to lower frequency. In the ligands and its complexes, this bands are occurs at 3400-3500^{cm-1} which excludes the possibility of hydrogen bonding. [21]

The purity and crystallinity of the synthesized nanoparticles were examined using XRD pattern. All the diffraction peaks are absolutely matched with the standard spectrum JCPDS number 96-901-6058 in the case of figure (1), JCPDS number 96-900-4180 in the case of figure (2).Diffraction peaks corresponding to the impurity were not found in the XRD patterns, confirming the high purity of the synthesized products. The average crystallite size is calculated by X-ray Diffraction, line broadening using the Scherrer formula.

$D = K\alpha / B \cos\theta$

- D=Crystalline size
- K=0.9 scherrer constant
- α=wavelength of X rays (1.5406 A° unit)
- θ =diffraction angle of the peak
- B=full width half maximum of the peak

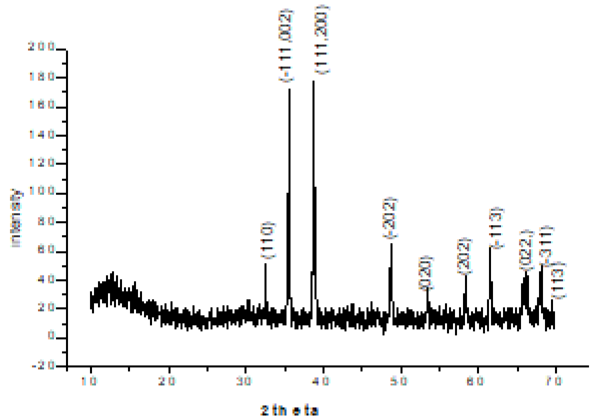

Figure 1: XRD pattern of CuO nanoparticles from Cu (VATSC)

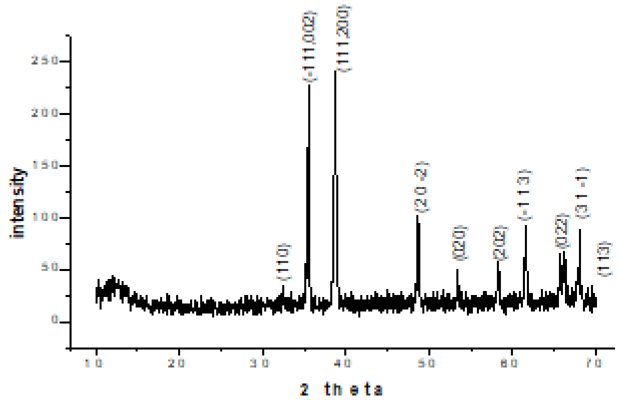

Figure 2: XRD pattern of CuO nanoparticles fromCu(SALTSC)

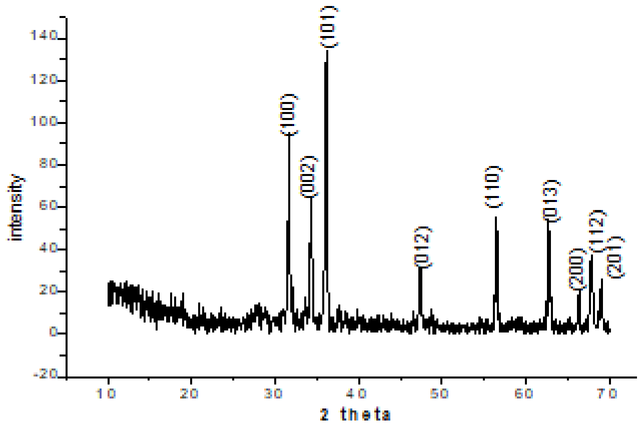

Figure 3: XRD pattern of ZnO nanoparticles from Zn (VANTSC)

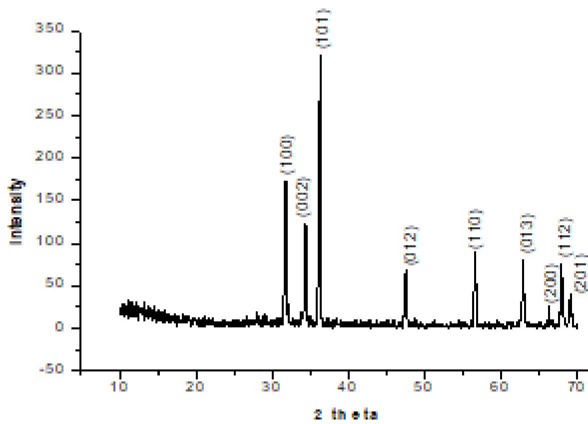

Figure 4: XRD pattern of ZnO nanoparticles from Zn (SALTSC)

Antibacterial Studies

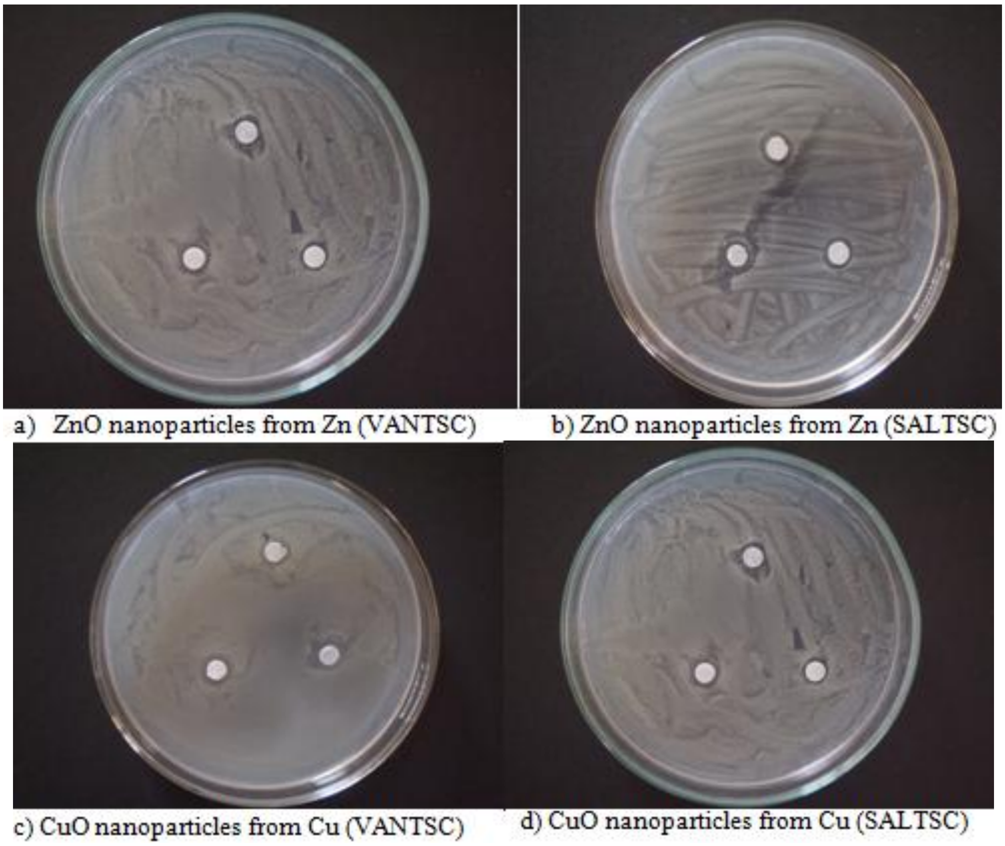

Figure 5: Antibacterial activity of nanoparticles

Different concentrations of the samples were tested by dispersing them in dimethyl sulphoxide (DMSO) and the results are shown in figure 5. Results shows(Table 1) that when the concentration of nanoparticles increases antibacterial activity increases and the zone of inhibition has been found to be maximum for 75 µg/mL. all the prepared nanoparticles were shown antibacterial activity Among the prepared nanoparticles ZnO nanoparticles from Zn(SALTSC) shows more activity.

Table 1: Antibacterial activity of nanoparticles

Sample name	25 µg/ml	50 µg/ml	75 µg/ml
ZnO from Zn (VANTSC)	+	+	++
ZnO from Zn(SALTSC)	++	++	+++
CuO from Cu (VANTSC)	+	+	++
CuO from Cu(SALTSC)	+	+	++

Key to symbols
Highly active = +++(inhibition zone>10
Moderately active=++(inhibition zone 7-10)
Slightly active=++(inhibition zone 4-6)

4. Conclusion

Calcination of synthesized complexes were achieved corresponded CuO and ZnO nanoparticles. Copper Oxides and Zinc Oxides having average Size of 30-35nm have been successfully prepared by solid-state thermal decomposition method. Antibacterial studies shows ZnO nanoparticles from Zn(SALTSC) shows good antibacterialpotential.

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