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Synthesis of CuO nanoparticles from a new nano-structured copper(II) Schiff base complex by ultrasonic and solvothermal methods: structural, thermal and antibacterial studies

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ABSTRACT

*Two new Cu(II) Schiff base complexes, $[Cu(L)_2](ClO_4)_2$ (**1**) and $[Cu(L)_2NO_3]NO_3$ (**2**) of Schiff base ligand (L = 4,5,9,13,14-pentaaza-benzo[b] triphenylen) were synthesized and characterized by elemental analyses (CHN), FT-IR, Uv–Vis and conductivity measurements. Nano-structure compounds of the complex (**1**) were prepared by sonochemical (**3**) and solvothermal (**4**) methods. The new nano-structures were characterized by scanning electron microscopy, X-ray powder diffraction, IR spectroscopy and elemental analyses. The thermal stability of bulk complexes and nano-sized particles (**3** and **4**) were studied by thermal gravimetric (TGA) and differential scanning calorimetry (DSC) analyses. CuO nano-particles were obtained by calcination of the nano-structure complexes at 450 ºC. The comparative electrochemical studies show that the copper complexes give quasi-reversible reduction process in CH_3CN solution. The free Schiff base and its metal complexes have been screened for antibacterial activities and the result show that the metal complexes are more active than its free Schiff base ligand.*

Keywords: Copper complex; Schiff base; Solvothermal; Copper nanooxide; Antibacterial.

INTRODUCTION

Schiff bases are considered as a very important class of organic compounds which have wide application in many biological aspects. Some Schiff bases were reported to posses antibacterial, antifungal and antitumor activities [1-2]. The transition metal complexes with Schiff bases as

ligands are of paramount scientific interest, due to their multiple implications [3].These complexes play important role in the developing of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures as well as for liquid-crystal technology [4-5]. 1,10-Phenanthroline-5,6-dione (pdon) is a versatile ligand for the assembly of metal organic materials. There are several reasons. It can be used directly as a bis-chelating ligand, and be prepared starting from an already complexed phenanthroline. The diketone functionality can also easily be transformed to other chelating groups such as a diamine or a dioxime. Moreover, it is also a versatile organic linker that can form bridges through amine condensation or a combination of coordination and condensation [6]. 1,10-Phenanthroline-5,6-dione is a chelate ligand containing an o-quinoid moiety which has many interesting characteristics. Many condensation reactions between an o-quinoid moiety of pdon and a diamine group have been reported [7]. 5,6-Diamino-1,10- phenanthroline (phen-diamine) is particularly important in that it can either directly bridge two metal centers or be condensed with a variety of ortho-quinones to form addition derivatives. For example, the useful bridging ligand, tetrapyrido[3,2-a:2',3",2"-h:2'",3'"-j]phenazine (tpphz), is readily formed upon condensation of phen-diamine with 1,10-phenanthroline-5,6-dione (phen-dione) [8]. These reactions are important in biological systems because they are crucial steps in biosynthesis, cellular biochemistry, metabolism, pharmacology and medicine. Copper Schiff base complexes are amongst the most versatile catalysts known for oxygenation reactions. The role played by copper ions in the active sites of a large number of metalloproteins has stimulated efforts to design and characterize copper complexes as models for a better understanding of biological systems. [9]. Macroscopic properties of materials strongly depend on both the size and the morphologies of the microscopic particles they are made up from. This is especially true for materials with morphological features smaller than a micron in at least one dimension, which is commonly called nano-scale materials, or simply nanomaterials. In these materials the ratio of surface area to volume is vastly increased when compared to compounds with larger grain sizes and quantum mechanical effects such as the ‘‘quantum size effect” begin to play a significant role. These effects only play a minor role when going from macro to micro dimensions, but become increasingly important when reaching the nanometer size range [10]. Thus synthesis and characterization of nano-structures with different particle sizes and morphologies are very important both from the viewpoint of basic science as well as for technological applications [11]. Nanoparticles have attracted great interest in recent years because of their unique chemical and physical properties, which are different from those of either the bulk materials or single atoms. Nano-materials have potential applications in optoelectronics, catalysis, and ceramics and so on [12]. Among these materials metal oxide nanoparticles are of technological importance for solar cells, chemical sensors and liquid crystal displays [12-14]. CuO, as an important p-type semiconductor with a narrow band gap (1.4 eV), has received great attention owing to its important properties and widespread applications [12]. Metal complexes built from metal ions and polydentate organic ligands have been grown rapidly in recent years owing to their potential applications. So far, however, the studies on the syntheses of nano- or microscaled structures with metal complexes as precursors have been less reported. Especially, much fewer studies on the preparation of CuO nanomaterials have been performed on copper complexes. The way using copper complexes as precursors may be helpful to manipulate the purity and morphology, and optimize the properties of CuO nanomaterial [12]. Due to the outbreak of the infectious diseases caused by different pathogenic bacteria, the scientists are searching for new antibacterial agents. In the present scenario, nanoscale materials have emerged up as novel antimicrobial agents

owing to their high surface area to volume ratio and the unique chemical and physical properties [15-16]. In recent years, the use of inorganic antimicrobial agents has been attracted interest for the control of microbes. The key advantages of inorganic antimicrobial agents are improved safety and stability, as compared with organic antimicrobial agents [16-17]. This work deals with the synthesis and characterizations of new copper (II) complexes of Schiff base ligand (4,5,9,13,14-pentaaza-benzo[b] triphenylen). Nano-sized particles as well as bulk compounds were prepared using sonochemical and solvothermal methods. The antibacterial activity of Schiff base and its copper (II) complexes are reported against the three gram-positive bacteria: *Streptococcus pyogenes (RTCC 1949), Staphylococcus aureus (RTCC 1113)* and *Bacillus anthracis (RTCC 1036)*. Microorganisms were cultured on Muller- Hinton agar medium.

EXPERIMENTAL SECTION

Material and Physical mesurments

All reagents and olefins were purchased from Merck and Fluka and used as received without further purification. Solvents used for reactions were purified and dried by conventional method [18]. Copper (II) perchlorate hexahydrate were purchased from Aldrich. 1, 10-Phenanthroline-5, 6-dione was prepared according to the reported method [19]. The Schiff base ligand (4,5,9,13,14-pentaaza-benzo[b] triphenylen) was synthesized according to the literature [20]. Caution: The perchlorate salts reported here are potentially explosive and, therefore, should be handled with care.

Elemental analyses (carbon, hydrogen and nitrogen) for the compounds were determined with an Elementar CHN Analyzer Vario El III. Infrared (FTIR) spectra were recorded using KBr discs on a Shimadzu FT-IR model Prestige 21 spectrometer. Melting points were determined using an electrothermal apparatus and were uncorrected. The UV-Vis spectra in 200-900 nm range were obtained in CH_3CN on a Perkin-Elmer lambda 25 spectrophotometer. The Conductivity measurements were carried out in acetonitrile in room temperature using a Metrohm conductometer instrument. TGA were carried out on a Mettler-Toledo TGA 851e at a heating rate of 10 °$Cmin^{-1}$ under a nitrogen atmosphere. The DSC thermograms of the compounds were obtained on a Mettler-Toledo DSC 822e module, which was calibrated with indium metal (T = 156.6 ± 0.3, ΔH = 28.45 ± 0.61 Jg^{-1}). Samples of 2-5.8 mg in solid form were placed in aluminium pans (40 μl) with a pierced lid, and heated or cooled at a scan rate of 10 °$Cmin^{-1}$ under nitrogen flow. Cyclic voltammmograms (CVs) were obtained using an Autolab modular electrochemical system (Ecochimie, Ulterecht, The Netherlands) equipped with a PGSTAT 20 module and driven by GPES (Ecochimie) in conjunction with a three- electrode system and a personal computer for data storage and processing. An Ag/Ag Cl (Saturated KCl)/ 3M KCl reference electrode, a Pt wire (counter electrode) and a glassy carbon working electrode, (Metrohm 0.0314 cm^2) were employed for the electrochemical studies. Voltammetric measurements were performed at room temperature in acteonitrile solution with 0.1 M tetrabutylammonium perchlorate as the supporting electrolyte. An ultrasonic generator (Dr. Hielscher UP400 S ultrasonic processor) equipped with an H22 sonotrode with diameter 22 mm, operating at 24 kHz with a maximum power output of 400 W, was used for the ultrasonic irradiation. The ultrasonic generator automatically adjusts the power level. X-ray powder diffraction (XRD) measurements were performed using a Philips diffractometer manufactured by

X'pert with monochromatized CuKα radiation. The nano-sized complexes were characterized with a scanning electron microscope (SEM) (Philips XL 30) with gold coating.

Preparation of copper complexes

General procedure: To an ethanol solution (5 mL) of metal salt (copper perchlorate or nitrate) (0.5 mmol), an ethanol solution (10 mL) of L (0.283 g, 1 mmol) was added. The resulting solution was refluxed for 2 h. The resultant colored solution was left at room temperature. The product was removed by filtration, washed with cooled absolute ethanol and recrystalized from methanol or acetonitrile and dried in vacuo.

$[Cu(L)_2](ClO_4)_2$ (1)

Yield: 0.22 g (53%). Anal. Found for $C_{34}H_{18}CuCl_2N_{10}O_8$: C, 49.37. H, 2.24. N, 16.95. Calcd: C, 49.29. H, 2.18. N, 16.90. m.p: 267 ˚C. FTIR (KBr disc): 3079, 1645, 1610, 1586, 1080, 625 cm^{-1}. Λ= 252 µS. UV–Vis (λ_{max}/nm): 698, 372, 357, 306, 296, 272.

$[Cu(L)_2NO_3]NO_3$ (2)

Yield: 0.15 g (58%). m.p: 247˚C. CHN. Found for $C_{34}H_{18}CuN_{12}O_6$: C. 54.20, H. 2.45, N. 22.33. Calcd: C. 54.15, H. 2.41, N. 22.28. IR (KBr): 3076, 1655, 1608, 1588, 1494, 1360, 1112, 820 cm^{-1}. Λ= 110 µS. UV–Vis (λ_{max}/nm): 716, 373, 356, 306, 296, 272.

Preparation of $[Cu(L)_2](ClO_4)_2$ (3) nanoparticles by sonochemistry method

10 ml of a 0.1 M solution of $Cu(ClO_4).6H_2O$ in EtOH were positioned in a high-density ultrasonic probe, operating at 24 kHz with a maximum power output of 400 W. In to this solution 10 ml of a 0.2 M solution of the ligand L was added dropwise. The obtained precipitates were filtered off, washed with methanol and then dried in air. Anal. Found for $C_{34}H_{18}CuCl_2N_{10}O_8$: C, 49.34. H, 2.24. N, 16.94. Calcd: C, 49.29. H, 2.18. N, 16.90. m.p: 267 ºC. IR (KBr disc): 3078, 1645, 1610, 1586, 1080, 625 cm^{-1}.

Preparation of $[Cu(L)_2](ClO_4)_2$ (4) nanorods by solvothermal method

$Cu(ClO_4)_2.6H_2O$ (0.185 g, 0.5 mmol) and ligand (L) (0.283 g, 1 mmol) were dissolved in 15 ml EtOH (15 ml). The solution was charged into a Teflon-lined stainless steel autoclave and heated at 150°C for 24h. After the autoclave was cooled immediately to room temperature, the product was filtered and dried and characterized. Anal. Found for $C_{34}H_{18}CuCl_2N_{10}O_8$: C, 49.35. H, 2.23. N, 16.96. Calcd: C, 49.29. H, 2.18. N, 16.90. m.p: 267 ºC. IR (KBr disc): 3078, 1645, 1610, 1586, 1080, 625 cm^{-1}.

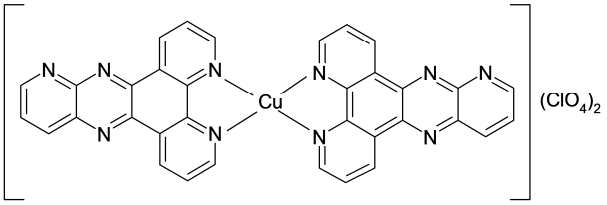
Antibacterial activity test of ligand and complexes

The in vitro activity test was carried out using the well method [21]. The potency of components was determined against the three Gram-positive bacteria: *Streptococcus pyogenes* (RITCC 1949), *Staphylococcus aureus* (RITCC 1113) and *Bacillus anthracis* (RITCC 1036). Microorganisms (obtained from enrichment culture of the microorganisms in 1 ml Muller-Hinton broth, incubated at 37 °C for 12 h) were cultured on Muller- Hinton agar medium. The inhibitory activity was compared with that of standard antibiotic, such as gentamicine (10 µg). After drilling wells on medium using a 6 mm cork borer 100 µL of solution from different compounds were poured into each well. The plates were incubated at 37 °C overnight. The diameter of the inhibition zone was measured to the nearest millimeter. Each test was carried out

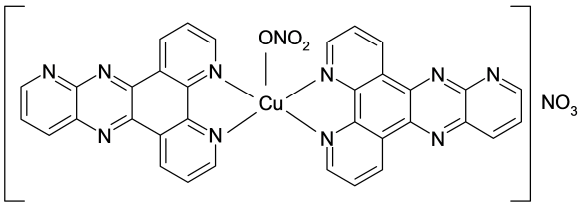
in triplicate and the average was calculated for inhibition zone diameters. A blank containing only methanol showed no inhibition in a preliminary test. The micro-dilution broth susceptibility assay was used for the evaluation of minimal inhibitory concentration (MIC). After incubation at 37 °C for 24 h the first tube without turbidity was determined as the minimal inhibitory concentration (MIC).

RESULTS AND DISCUSSION

The ligand 4,5,9,13,14-pentaaza-benzo[b] triphenylen (L), has been obtained by [1+1] cyclocondensation between 1,10-phenanthroline-5,6-dione and 2,3-diaminopyridine. The ligand L readily reacts with $Cu(X)_2.nH_2O$ (X= ClO_4, NO_3, n= 6 and 3, respectively) in ethanol to produce the corresponded copper (II) complexes of (**1**) and (**2**) (Fig. 1). The complexes were characterized by elemental analyses (CHN), UV-Vis, FTIR spectroscopy and molar conductivity. These complexes were found to be fairly soluble in methanol, acetonitrile, DMF and DMSO and display good stability in the air at room temperature.



(**1**)



(**2**)

Figure 1: Suggested structure of the Cu (II) complexes

The infrared spectra of free ligand and their complexes recorded in the region 4000-400 cm^{-1}. The spectra data for the complexes confirm the cyclic nature of the ligands in their metal complexes and show a band at 1646–1657 cm^{-1}, attributable to the imine groups, and no bands due to ν(C=O) vibrations. The spectra exhibit medium bands at 1609–1612 cm^{-1} as expected for the ring vibrations of the coordinated phenanthroline and 1585-1588 cm^{-1} vibration as expected to pyridine [22]. The FT-IR spectra of all complexes compared with of the ligand, indicates that the ν(C=N) band at 1602 cm^{-1} is shifted to higher frequency by 7-10 cm^{-1} in the complexes, indicating that the ligand are coordinated to the metal ions through the nitrogen atom of the phenanthroline group [22]. The IR spectra for the perchlorate complexes showed absorptions attributable to the perchlorate ions at approximately 1085 and 625 cm^{-1} [23]. The lack of splitting of these bands suggests that the perchlorate anions are not coordinated [22]. Complex (**2**) show bands in 1494 cm^{-1} (ν_1), 1360 cm^{-1} (ν_5), 1112 cm^{-1} (ν_2), and 820 cm^{-1} (ν_6) regions indicating the coordination of nitrate group [24,25]. The separation of ~134 cm^{-1} between ν_1 & ν_5 indicates unidentate coordinating nature of nitrate group [25]. In the complex (**2**) an intense band at 1384

cm^{-1} attributable to ionic nitrate, is also present [26]. The molar conductivity data at room temperature show 1:2 and 1:1 electrolytic natures for complex (**1**) and (**2**), respectively [27].

The absorption spectroscopic behaviour of compounds (**1**) and (**2**) were studied in CH_3CN. All of the complexes exhibit three absorption bands at 250–270 nm and 320–380 nm, due to the ligand centred π–π* transitions or charge transfer (CT) transitions [28]. The appearance of broad absorption band at 698 in the spectrum of the complex (**1**) which are assigned to d-d transitions, suggests that the coordination geometry at the metal ion could be distorted from square planer [29]. In complex (**2**) observed absorption band at 716 nm can be assigned to square pyramidal geometry around the metal centre [24, 30]. Nano-structures of **(1)** were obtained by both ultrasonic irradiation (**3**) and solvothermal (**4**) in an ethanolic solution, respectively. IR spectra of the nano-structures are indistinguishable. Obtained XRD data show that XRD pattern of bulk compound (**1)** is the same in comparison with the XRD pattern of complex of **(3)** and **(4)** prepared by the sonochemical and solvothermal methods, respectively. The obtained data indicates that the compounds obtained by both hydrothermal and sonochemical processes as bulked complex are an amorphous phase. The size of the particles is 48-56 nm, which is in agreement with that observed by scanning electron microscopy (Fig. 2).

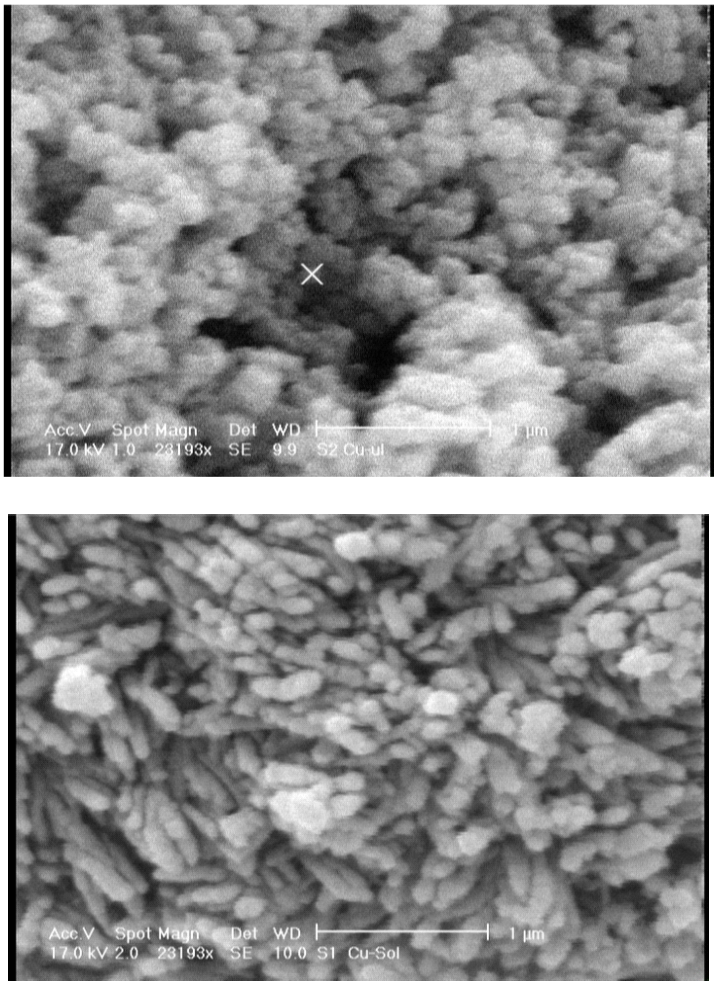


Figure 2: SEM images of nanoparticles (3) (top) and nanorods (4) (bottom) as produced by ultrasound irradiation and solvothermal respectively

Thermal decomposition of the nano-sized particles of **(1)** in air at 450 °C produced CuO nano-structures. Fig. 3 shows the SEM image of the CuO nano-structures.

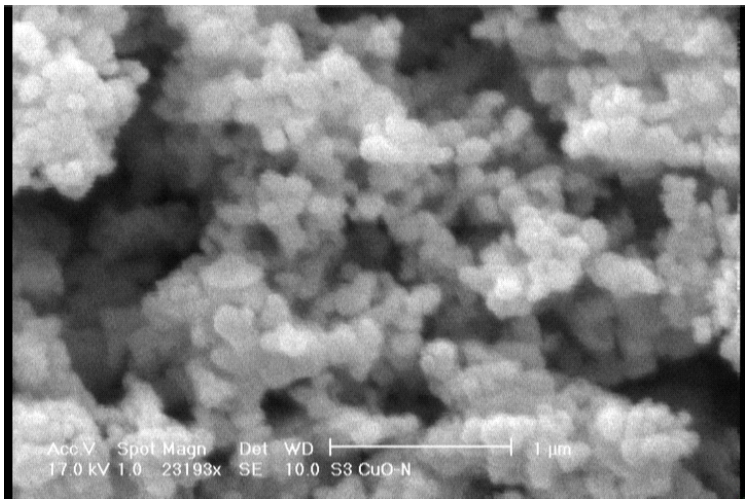


Figure 3: SEM image of CuO nanoparticles prepared by solvothermal

Fig. 4 shows the X-ray diffraction (XRD) pattern of the prepared CuO nano-structures. CuO nano-structures have shown the most crystallinity because of the existence of sharp peaks in the XRD pattern. The phase purity of the as prepared CuO nano-structures is completely obvious and all diffraction peaks are perfectly indexed to the monoclinic CuO phase with the lattice constants comparable to the reported data (JCPDS 01-1117). No characteristic peaks of impurities are detected in the XRD pattern. The broadening of the peaks indicated that the particles were of nanometer scale.

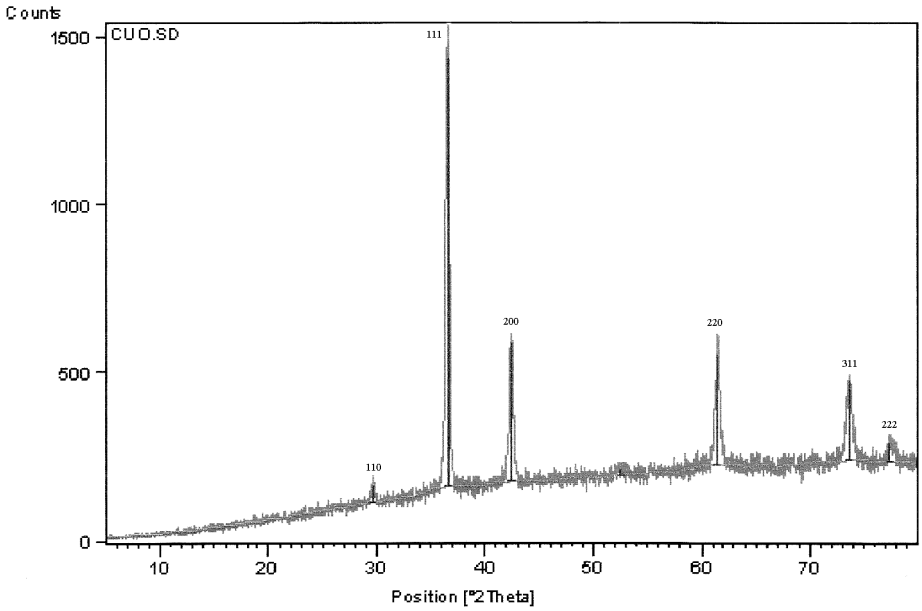


Figure 4: XRD pattern CuO nanoparticles prepared by solvothermal

Cyclic voltammograms (CVs) of the complexes (**1**) and (**2**) were recorded in acetonitrile and with 0.1 M tetrabutylammonium perchlorate as supporting electrolyte in the potential range −2 to +0.1V. Typical CVs of (**1)** are shown in Fig. 5.

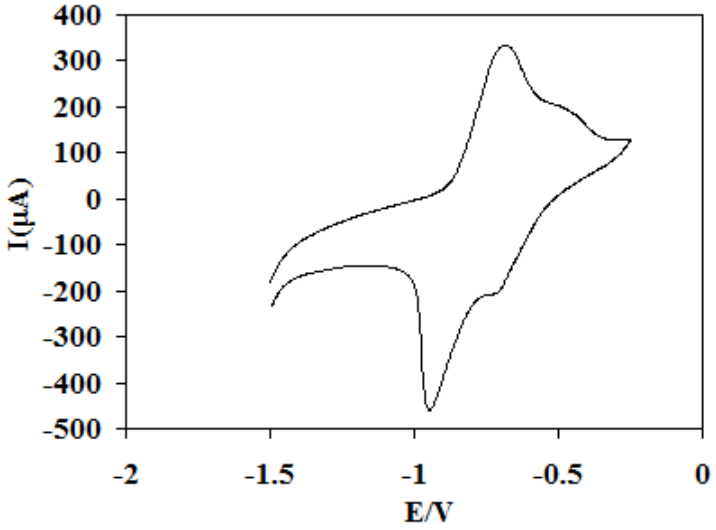


Figure 5: Cyclic voltammograms of a 10^{-3} M solution of (1) in acetonitrile, in presence of 0.1 M tetrabutylammonium perchlorate (TBAP), using working electrode: glassy-carbon, reference electrode: Ag/AgCl; auxiliary electrode: platinum wire, scan rate 100 mV.

Upon sweeping the voltage to oxidizing potentials the complex (**1**) undergoes one electron quasi-reversible (ΔE_p = 280 mV) redox process (Table 1) which can be ascribed to the Cu^{II}/Cu^{III} couple (E_a= -0.45 V, E_c= -0.73 V). Anodic and cathodic peak (E_a= -0.71 V and E_c= -0.95 V) is observed at nearly the same potential value as the corresponding ligand and may be due to irreversible redox process of the ligand.

Table 1: Electrochemical data for copper (II) complexes in CH_3CN solution

Complex	E_{pa}(V)	E_{pc}(V)	ΔE_p[a](mV)	i_{pa}/i_{pc}	$E_{1/2}$[b]
(1)	-0.453	-0.733	280	0.89	-0.59
(2)	-	-0.660	-	-	-
(3)	-0.447	-0.725	278	0.89	-0.59
(4)	-0.450	-0.729	279	0.89	-0.59

[a] *$\Delta E_p = E_{pc}-E_{pa}$ at scan rate 100 mV*

[b] *Data from cyclic voltammetric measurements; $E_{1/2}$ is calculated as average of anodic (E_{pa}) and cathodic (E_{pc}) peak potentials = E1/2= 1/2 ($E_{pa}+E_{pc}$)*

Obviously the separation between E_p increases as the scan rate increases and this is characteristic of a quasi-reversible system. There is a linear relationship between the cathodic and anodic peaks currents and the square root of the scan rate ($\nu^{1/2}$) in the 100-450 mVs^{-1} range (Fig. 6). This behavior is typical for an electron transfer process controlled by diffusion. The complex **(2)** exhibited one irreversible reduction peak related to Cu^{II}/Cu^{I} reduction (E_c= -0.66 V) in addition to the ligand peaks.

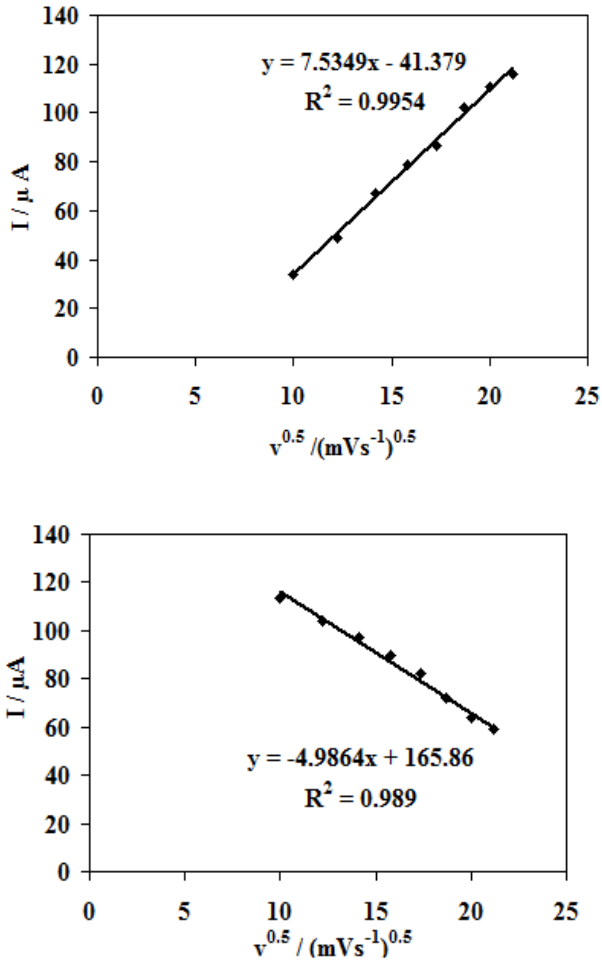


Figure 6: Plot of anodic and cathodic currents vs. the square root of sweep rate ($v^{1/2}$) for (1) complex.

To examine the thermal stability of the nano-sized complexes and the corresponded bulk complex, thermal gravimetric analyses (TGA) and differential scanning calorimetry (DSC) analyses were carried out between 25 and 850 °C in a static atmosphere of nitrogen. Enthalpy changes and decomposition temperatures of Schiff base ligand and related copper (II) complexes are tabulated in Table 2. The TGA data indicate that the complex (**1**) start decomposition at 285 °C. There is no mass loss up to 200°C, indicating that either water or solvent molecules are absent in this complex. On the base of DSC obtained data, nano-sized complex (**3**) start to decompose at 232.48 °C. Detectable decomposition of the nano-complex of $[Cu(L)_2](ClO_4)_2$ starts about 45 degree (Table 2) earlier than that of its bulk counterpart, probably due to the much higher surface to volume ratio of the nano-sized particles as more heat is needed to annihilate the lattices of the bulk compounds.

Antibacterial activities (zone of growth inhibition and minimal inhibitory concentrations) of Schiff base ligand, their related complexes and gentamicine (as a standard compound) are shown in Table 3. The organisms used in the present investigation included *Streptococcus pyogenes*

(RITCC 1940), *Staphylococcus aureus* (RITCC 1885) and *Bacillus anthracis* (RITCC 1036) as gram-positive bacteria.

Table 2: Thermoanalytical (enthalpy changes and decomposition temperatures) results of free Schiff base ligand and related Cu(II) complex

Complex	T^a(C°)	$\Delta H^a(Jg^{-1})$	$T_d{}^b$(°C)
(1)	277.95	28.51	285
(2)	309.06	63.63	315
(3)	232.48	53.64	240
(4)	233.06	59.23	242

[a] Data obtained from first DSC cycle.
[b] Data obtained from TGA; 10 °C min^{-1} under N_2 gas

The obtained data reflect that the free Schiff base has moderate activity in comparison with *S. pyogenes* and *S. aureus* and good activity towards *B. anthracis*. The complex **(1)** is moderately active (inhibitory zones >15mm) against two gram-positive bacteria. The collected results in Table 3 indicated that the complex **(1)** is weakly active against *S. pyogenes* (inhibitory zones < 15 mm). The complex **(2)** has better activity than **(1)** against three tested bacteria. The antibacterial activities of both nano complexes **(3-4)** are similar with complex **(2)**; this is probably because of diminishing of the size of particle in complexes **(3-4)** in comparison with complex **(1)**.

Table 3: Antibacterial activities (zone of growth inhibition and minimal inhibitory concentrations) of Schiff base ligand and copper (II) complexes and gentamicine (as a standard compound)

Method	Main compounds	Microorganism		
		Stereptococcs Pyogenes (+)	*Bacillus anthrcis (+)*	*Staphylococcus aureus (+)*
Growth Inhibitory zone [mm]	L	16	25	17
	(1)	13	20	20
	(2)	18	25	25
	(3)	18	25	25
	(4)	18	25	25
Standard	Gentamicine	13	32	13
Minimum inhibitory concentration (mg/ml)(MIC)	L	25	12.5	25
	(1)	50	25	25
	(2)	25	12.5	12.5
	(3)	25	12.5	12.5
	(4)	25	12.5	12.5

The antibacterial activities values for the free Schiff base ligand are lower than those found for the complex **(2)**. It appears that the coordination of metal ion to Schiff base ligand increases the antibacterial activity of the corresponded Schiff base. The activity of complex **(2)** may be explained on the basis of chelation theory; chelation reduces the polarity of the metal atom mainly because of partial sharing of its positive charge with the donor groups and possible π electron delocalization within the whole chelate ring. Also, chelation increases the lipophelic nature of the central atom which subsequently favors its permeation through the lipid layer of the cell membrane [31]. The antibacterial results showed that all of the synthesized compounds

exhibit most activity against the *S. aureus* in comparison with standard antibiotic and among the complexes, the complexes **(2-4)** were found to be relatively active towards three tested gram-positive bacteria. The quantitative assays gave MIC values in the region 12.5-50 $mgml^{-1}$ (Table 3), that confirmed the above obtained results.

CONCLUSION

Cu(II) forms complexes of composition $[Cu(L)_2](ClO_4)_2$ (**1**) and $[Cu(L)_2NO_3]NO_3$ (**2**) where L is the Schiff base ligand of 4,5,9,13,14-pentaaza-benzo[b] triphenylen. Nano-sized of complexes **(3)** and **(4)** were obtained by both solvothermal and ultrasonic irradiation, respectively and characterized by common physicochemical as well as XRD and SEM techniques. Calcination under air atmosphere of complex (**1**) produces nano-sized particles of CuO. The antimicrobial activities show that the free Schiff base ligand has moderate activity against three gram positive bacteria. The complex (**2**) has good activity against three gram-positive bacteria while has moderate activity against *Streptococcus pyogenes*. The complex (**2**) shows better activity against all tested bacteria in comparison with free Schiff base ligand and gentamicine, except against *Bacillus anthracis* in which gentamicine shows relatively better activity. This study demonstrates that the coordination compounds may be suitable precursors for the preparation of nano-scale materials. The sonochemistry method may have some advantages such as: it takes place with shorter reaction times and produces better yield.

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