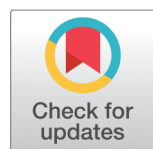


# Synthesis and photophysical study of divalent complexes of chelating Schiff base

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## ABSTRACT

**Background:** Schiff base compounds derivative from 1,2,4-triazole, and their transition metal complexes play an essential role in coordination and bioinorganic chemistry due to biological and industrial applications.

**Objectives:** This work aims to prepare and characterize 1,2,4-triazole Schiff base and its complexes with a theoretical study, photophysical properties, and surface morphology for these complexes.

**Methods:** 1,2,4-triazole Schiff base prepared by condensation reaction between 4-Amino-3-mercapto-5-phenyl-4H-1,2,4-triazole and 2-hydroxy-1-naphthaldehyde, then Schiff base reacted with  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Cu}^{2+}$  ions, the synthesized 1,2,4-triazole Schiff base, and its complexes were characterized by infrared spectra, magnetic susceptibility, conductivity measurements, photophysical properties, and surface morphology measured by atomic force microscopy. The practical results were reinforced with a theoretical study, using PM3 calculation and HyperChem program, for these Schiff base complexes.

**Results:** 1,2,4-triazole Schiff base act as a chelate ligand. The coordination has occurred through the oxygen of the phenolic group O-H and the nitrogen of the imine group  $\text{N}=\text{C}$  of Schiff base with divalent metal ions. Cobalt complex has a tetrahedral geometry, while the nickel and copper complexes have square planar geometries. The stability of all compounds was studied by calculating the energy gap by diffuse reflectance spectroscopy and theoretical calculations. Copper Schiff base is a more stable complex due to the lower value of the energy gap, and the copper Schiff base complex is more semiconductivity than the other complexes. Surface morphology, properties of chelating Schiff base ligand and its complexes measured by atomic force microscopy, cobalt Schiff base complex is higher roughness. The bond length of ( $-\text{C}=\text{N}-$ ), ( $-\text{C}-\text{O}-$ ), ( $\text{M}-\text{O}$ ), and ( $\text{M}-\text{N}$ ) are affected in the coordination with metal ions, the bond length of the square planar geometry more affected than tetrahedral geometry.

**Conclusions:** All compounds were prepared successfully, characterized, and photophysical properties were studied.

**Keywords** energy gap, morphology, triazole metal (II) complexes, triazole Schiff base

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## INTRODUCTION

Schiff bases are compounds prepared by Hugo Schiff via condensation of primary amines and carbonyl compounds; the products have an imine group.<sup>1</sup> The literature has many published types of research on Schiff base complexes containing 1,2,4-triazole moiety which have antifungal, anticancer, antibacterial, and anti-inflammatory like non-steroidal anti-inflammatory drugs so that it can use in the treatment of communicable diseases, Candida infections, and Blastomyces dermatitidis. The biological activity of Schiff base increases by the coordination with transition metal ions.<sup>2-5</sup> Besides, Schiff bases and its metal complexes act as anti-ureas agents, catalyze urea hydrolysis by ureases.<sup>6</sup> The industrial field of the Schiff base is used as corrosion inhibitors.<sup>7,8</sup>

Recent studies focus on the preparation of new transition metal complexes of 1,2,4-triazole Schiff bases with their biological activity. Schiff bases complexes of triazole exhibit better biological activity than the free triazole derivatives and the geometry of these complexes suggested by different spectral analysis.<sup>9</sup> The stable metal ions of the Schiff bases were synthesized and characterized by mass analysis, <sup>1</sup>H NMR, ultraviolet, infrared, and molar conductivity, the result of the antibacterial activity indicates that metal complexes show increases in activity compared to a free Schiff base ligand.<sup>10</sup> Schiff base complexes were synthesized and characterized by analytical spectroscopy, chelation of Schiff base with metal ions occurs through the deprotonated phenolic group and azomethine group; therefore, octahedral geometry was suggested. The effect of these complexes on antibacterial was studied and showed the activity increases with complexation.<sup>11</sup> Hexadentate Schiff base derivative from salicylaldehyde was reacted with Cr III, Fe III, Co III ions, and characterized by <sup>1</sup>H NMR, <sup>13</sup>CNMR spectra, infrared, electronic spectra, elemental analysis, thermal analysis, conductivity, and magnetic susceptibility measurements, the metal ion was coordinated with Schiff base ligand through the oxygen of phenol, the nitrogen of imine group and oxygen of ether atoms to suggest octahedral geometry. The biological activity of hexadentate Schiff base derivative with its complexes was measured.<sup>12</sup> The Schiff base derived from 1,2,4-triazole react with Cu<sup>2+</sup> and Zn<sup>2+</sup> ion using 1:2 (Metal: Ligand) mole ratio, These complexes were characterized by different chemical and physical technics so that Cu<sup>2+</sup> and Zn<sup>2+</sup> complexes have octahedral geometry, the antimicrobial activity of these complexes was measured.<sup>13</sup> The presence of a Schiff base containing the 1,2,4-triazole in polystyrene film showed enhancing photostability for polystyrene against irradiation by ultraviolet light (14). Co<sup>2+</sup>, Ni<sup>2+</sup>, Cu<sup>2+</sup>, and Zn<sup>2+</sup> complexes of 1,2,4-triazole Schiff bases were prepared using molar ratio 1:1 (ligand: metal), the structure of the Schiff bases and its metal complexes were characterized by different spectrometry, the ligand act as negative tridentate. From spectral studies and theoretical calculations the Co<sup>2+</sup>, Ni<sup>2+</sup> complexes have octahedral geometry, Cu<sup>2+</sup> complex has a square planar geometry, and tetrahedral geometry for Zn<sup>2+</sup> complex. Also, these metal complexes have good anticancer activity against human breast cancer cell line compared to the free ligand.<sup>14</sup>

This work aims to prepare and characterize the Schiff base derivative from 1,2,4-triazole, then the Schiff base reacted with divalent metal ions, as well as the work involves theoretical

study using PM3 calculation and HyperChem program, surface morphology, and photophysical properties of the prepared complexes.

## MATERIALS AND METHODS

### Chemicals and reagents

The chemicals used have high purity. Ethanol (98%),  $\text{CoCl}_2$  (99.9%),  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (99.8%), and  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (99.9%) were purchased from Sigma-Aldrich. Sodium acetate (98.9%) was used from Honeywell Fluka.

### Instrumentation

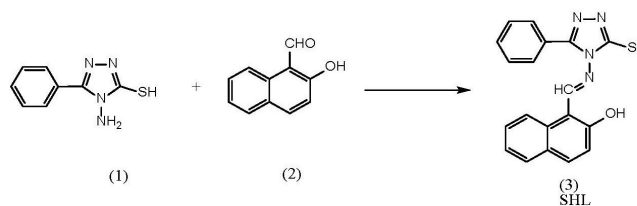
All compounds were characterized by FTIR ALPHA spectrophotometer (Bruker Optics, Model Alpha), magnetic susceptibility balance by Johnson Matthey, which can be used for describing paramagnetic and diamagnetic materials. Molar conductivity measurements were carried by corning conductivity meter 220. The photophysical properties were measured by diffuse reflectance spectroscopy (Avantes DH-S-BAL-2048 UV-Vis) in a wavelength range of 230-1100 nm. The Surface Morphology properties measured by Atomic Force Microscopy (AFM), AFM analysis was used to scan, prop microscopy Angstrom advanced AA 2000 and a model used was contact mode, under normal atmospheric conditions.

### Selection of solvents

In the solubility, ethanol was selected as the solvent for dissolving 1,2,4-triazole Schiff base, and its complexes.

### Preparation of 1,2,4-triazole Schiff base (SHL)

Schiff base ligand (SHL) was prepared by condensation reaction of 4-Amino-3-mercapto-5-phenyl-4H-1,2,4-triazole (**1**) (0.9g/ 0.01mol) with 2-hydroxy-1-naphthaldehyde (**2**) (1.8g/ 0.01mol) in ethanolic solution (100ml) 4-5 drops of glacial acetic acid was added in 250ml round bottom flask, the mixture was refluxed for 8 hrs. The resulting solution left to cool, then the precipitate was filtered, and washed with ethanol and dried, yellowish powder (**3**) was identified by FTIR analysis,<sup>15-17</sup> the synthesis of SHL was shown in Figure 1.



**Figure 1** Preparation of SHL

## Preparation of divalent metal complexes

A hot ethanolic solution of the SHL (0.69 g/0.02 mol), was added to the ethanolic solution (0.13g/ 0.23g/ 0.17g; 0.01 mol) of  $\text{CoCl}_2$ ;  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ;  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ , respectively. Then sodium acetate (0.5 g) was added to the mixture to adjust the pH of the solution, after that reflux the reaction for 4 hrs. Then pouring the mixture into the 100 ml distilled water to separate the product. Filter the suspended solid metal complex to collect it, washed with distilled water several times, and crystallization with a hot ethanol solution and place in the oven at  $50^\circ\text{C}$ .<sup>13</sup> The yield was 85%.

## RESULTS AND DISCUSSION

### Infrared spectra

The infrared frequencies of the SHL and its complexes are shown in Table 1. Schiff base has bands appear at  $2520.0\text{ cm}^{-1}$  and  $788.0\text{ cm}^{-1}$  due to  $\nu\text{S-H}$  and the  $\nu\text{C=S}$ , respectively, so that the tautomerism expected, this phenomena also appear in the spectra of the complexes that means thioamide group not involving in coordination with metal ions. The  $\nu\text{C=N}$  imine band appears at  $1618.33\text{ cm}^{-1}$  was shifted to a lower frequency in the SHL complexes; therefore, the coordination with metal ions through the nitrogen of the imine group occurred. Schiff base ligand has a  $\nu\text{O-H}$  band at  $3250.10\text{ cm}^{-1}$  due to the phenolic group, disappearing this band in SHL complexes, indicates the oxygen coordinated with the metal ion. Besides,  $\nu\text{C-O}$  stretching vibrations in the SHL complexes appear at  $1076.44\text{ cm}^{-1}$  shifted to longer wave numbers due to coordination. New bands at  $(598.91-500.93)\text{ cm}^{-1}$  for  $\nu\text{M-O}$  and  $(511.09-417.20)\text{ cm}^{-1}$  for  $\nu\text{M-N}$  appears in the lower wavenumber region.<sup>4,5,18</sup>

The magnetic moment of  $\text{Co d}^7$  was 4.62 B.M.; therefore, the suggested geometry was tetrahedral due to paramagnetic properties.  $\text{Ni d}^8$  has a magnetic moment zero due to diamagnetic properties; therefore, the square planar geometry was suggested.  $\text{Cu d}^9$  has a magnetic moment 1.67 B.M. so that square planar geometry was proposed,<sup>19</sup> the complexes showed non-electrolytic properties through the conductivity measurements using the DMF solvent, see Table 2.

**Table 1** Infrared bands of SHL and its metal complexes,  $\text{cm}^{-1}$ .

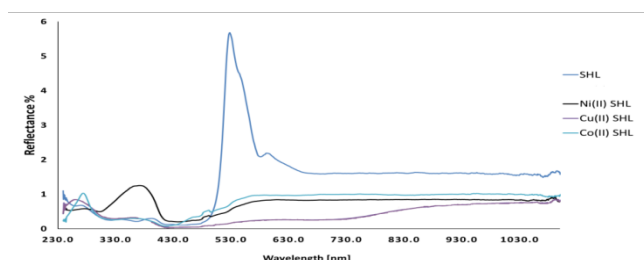
Compound	$\nu\text{O-H}$	$\nu\text{C=N}$	$\nu\text{C-O}$
SHL	3250.10	1618.33	1076.44
CoSHL	-	1574.50	1143.73
NiSHL	-	1575.80	1144.43
CuSHL	-	1573.44	1103.01

**Table 2** Physical data of SHL and c complexes.

Compound	Colour	Conductivity solvent $\mu\text{s/cm}$	DMF	Suggested formula	Magnetic moment, B.M.
SHL	Yellow	-		$\text{C}_{18}\text{H}_{14}\text{N}_4\text{SO}$	-
CoSHL	Greenish blue	12		$\text{Co C}_{36}\text{H}_{26}\text{N}_8 \text{S}_2\text{O}_2$	4.620
NiSHL	Light green	15		$\text{Ni C}_{36}\text{H}_{26}\text{N}_8 \text{S}_2\text{O}_2$	Zero
CuSHL	Green	19		$\text{Cu C}_{36}\text{H}_{26}\text{N}_8 \text{S}_2\text{O}_2$	1.68

## Reflectance and band energy measurement

The photophysical properties of SHL and its complexes were measured by diffuse reflectance spectroscopy DRS in the range 230–1100 nm, see Figure 2. The electrons transition from the low spin (valence band) to the high spin (conductive band) to measure the band energy of the prepared complexes.<sup>20–22</sup>

**Figure 2** Diffuse reflectance spectra of SHL and complexes

The conductive properties of the complexes measured the band gaps from diffuse reflectance spectra using Kubelka–Munk theory.<sup>23</sup> The plots of  $[F(R_\infty) / h\nu]^2$  versus photon energy ( $h\nu$ ) were used, and the intercept determines the band gaps value. Where  $R_\infty$  = sample reflection coefficient,  $\lambda$  = absorption wavelength,  $F(R)$ : function, and photon energy: (he), see Figure 3

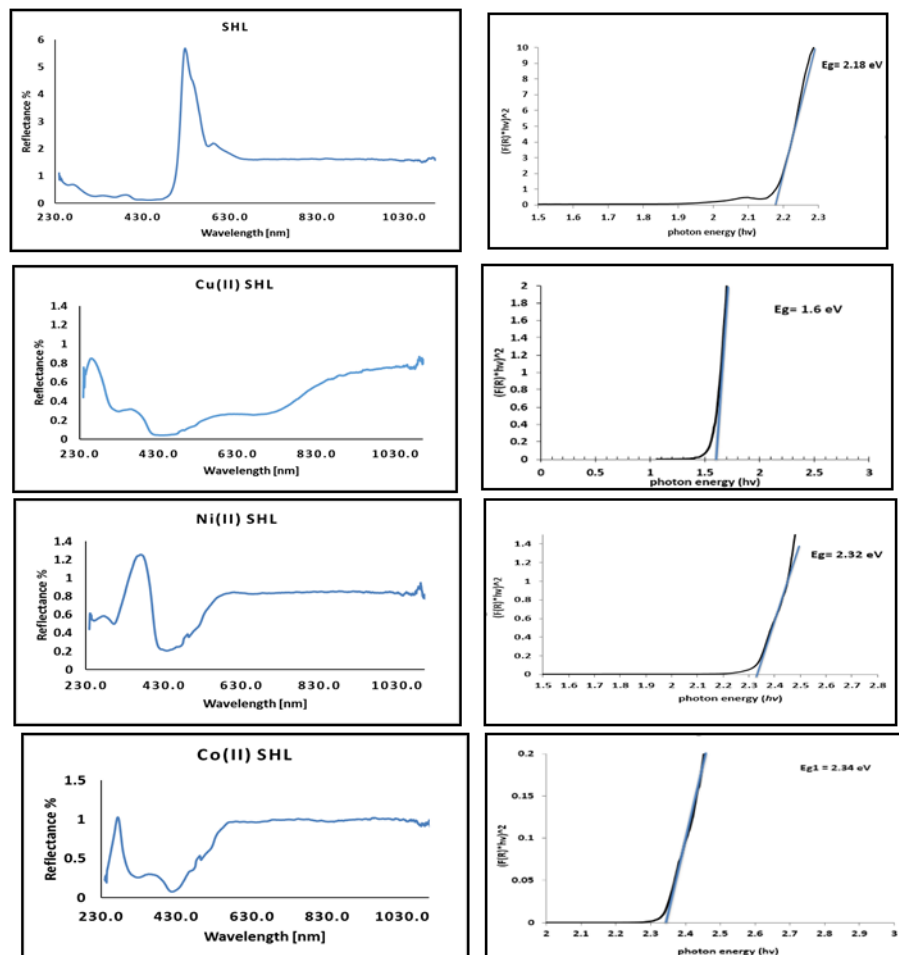
$$F = \frac{(1-R_\infty)^2}{2R_\infty} \dots (1)$$

$$\alpha h\nu = C_1(h\nu - E_g)^{1/2}$$

$$\alpha = F(R_\infty) \dots (2)$$

$$[F(R_\infty) / h\nu]^2 = C_2(h\nu - E_g) \dots (3)$$

$$h\nu = 1240/\lambda$$



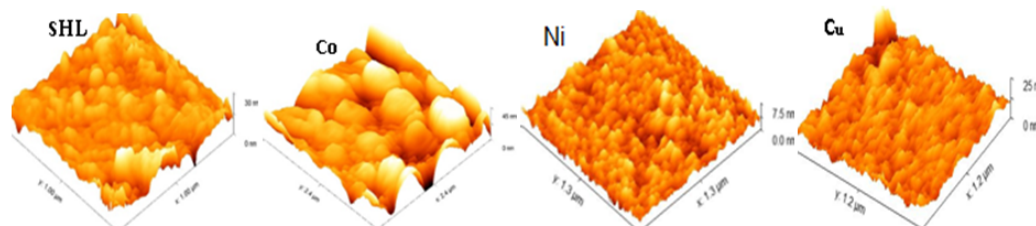
**Figure 3** Reflectance and band energy gap of SHL and complexes

The energy gap of SHL was 2.18 eV, the complexes with a square planar geometry the energy gap was 1.6 eV for CuSHL and 2.32 eV for NiSHL, while in tetrahedral geometry the energy gap was 2.34 eV for CoSHL, so it is a higher value compared with the other complexes; therefore the CoSHL complex will be less semiconductive. The CuSHL complex has the best semiconductive properties due to the small bandgap indicates to the easy electronic transitions from the HOMO to LUMO energy levels.<sup>24</sup>

### Surface morphology

The Surface morphology properties measured by atomic force microscopy AFM, the 3D image of the surface morphology for SHL, and its complexes, see Figure 4. The 3D image of the surface shows that the Schiff base was irregular particle distribution while the complexes have a regular homogenous distribution appearance and the partial size scale. More regular

homogenous distribution appearance in NiSHL complex and CoSHL particles form disk-like and more diameters. Topography data are shown in Table 3. The surface roughness of NiSHL becomes very smooth than SHL this leads to increase light scattering and reflectance, then a high energy gap, while CoSHL, topography properties are high.



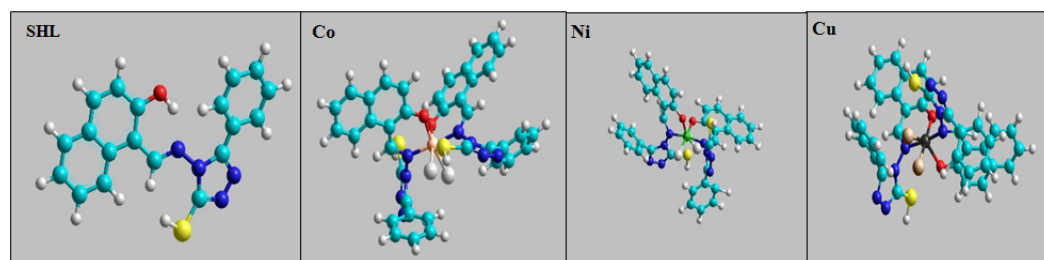
**Figure 4** Surface morphology, 3D image of SHL and complexes.

**Table 3** Topography properties of SHL and c mplexes.

Complex	RMS Surface Roughness(nm)	Average Diameter(nm)	Average Height(nm)	Average Size (nm <sup>2</sup> )
SHL	3.29	18.58	17.81	271.08
CoSHL	8.85	60.67	35.72	2890.50
NiSHL	0.835	12.10	7.08	114.93
CuSHL	2.34	23.40	14.50	429.90

## Theoretical studies

Hyper Chem 8.0.1 software and using semi-empirical calculations with the PM3 method to estimate the optimized molecular structure of the SHL and complexes, see Figure 5. All geometric structure is shown non-planar, and the coordination occurs between a metal with nitrogen and oxygen atoms.<sup>25</sup>



**Figure 5** Optimized structure balls and cylinder shape of SHL and its c complexes

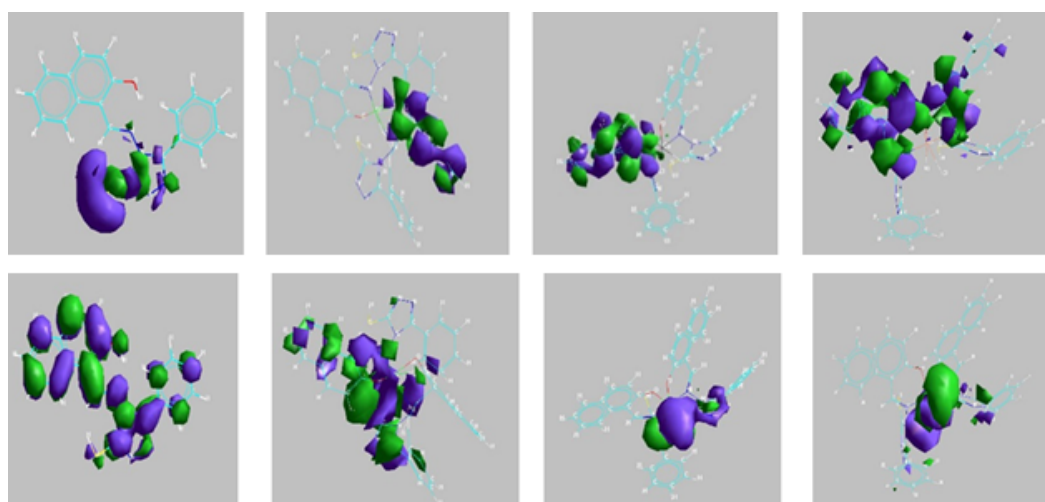
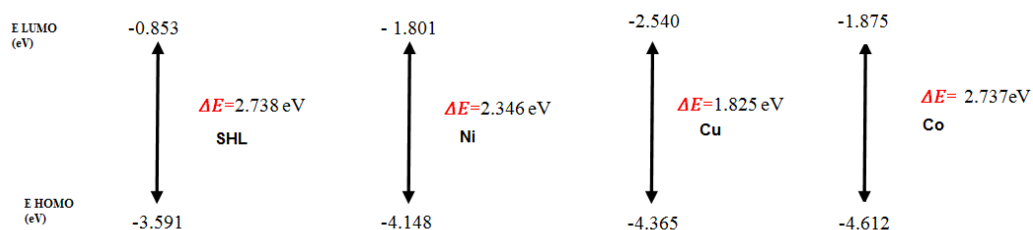




## Semi-conductive properties and electronic transition of molecular orbitals

The stability of the structures and electrical transport properties determined in the theoretical study as an energy gap, which is the difference between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO), (i.e.  $E_{gap} = E_{LUMO} - E_{HOMO}$ ).

The stability of the SHL and the complexes were shown when the energy gap calculation lower value that means the higher stability complex formed are shown in Figure 6, CoSHL complex has the highest energy gap ( $\Delta E = 2.737$  eV) that means the electron stay in a ground state level (lower energy) more than transition to the excited state (high energy). CuSHL complex was the energy gap ( $\Delta E = 1.825$  eV) and become more semi-conductive than other complexes.<sup>26</sup>

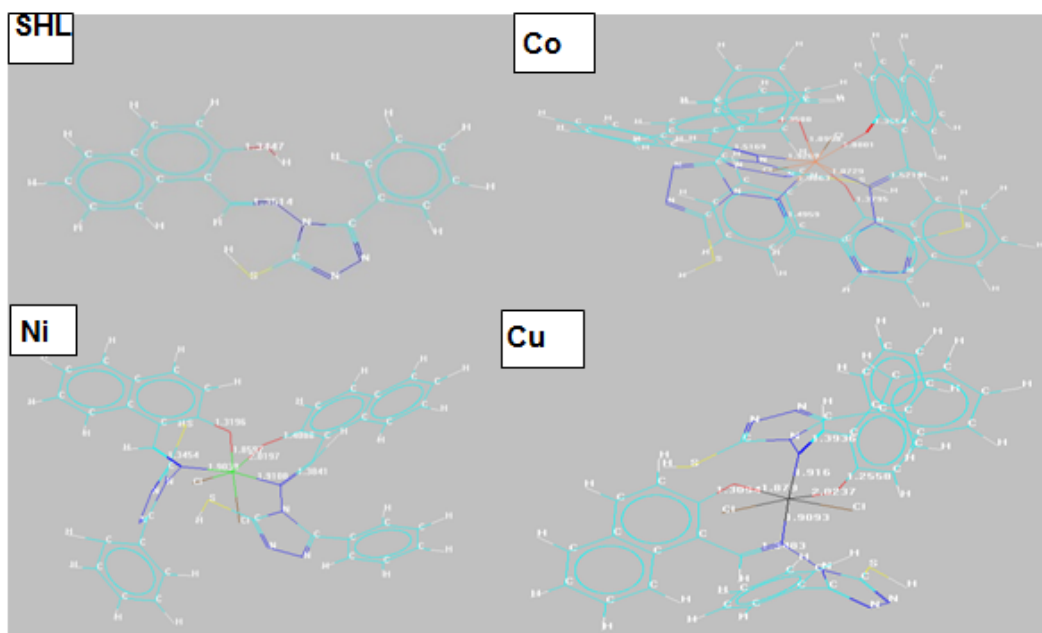


**Figure 6** Frontier molecular orbitals of structures of SHL and its complexes



## The effect of the coordination on bond length

The theoretical study and the practical results from FTIR spectra show that the coordination occurs between metal ion and oxygen of phenolic group and nitrogen of imine group (-C=N-) of the Schiff base ligand and the bond length effect as the metal used and the structural form. Table 4 and Figure 7 show that effected. The CuSHL the bond length of (-C=N-), (-C-O-) increases when compared with a free ligand.



**Figure 7** Some bonds lengths of Schiff base and complexes

**Table 4** Some bonds lengths of SHL and its complexes

Compound	Metal-O	Metal -N	C=N	C-O
SHL	-	-	1.3514	1.3447
NiSHL	2.0197 1.8597	1.9108 1.9039	1.3841 1.3454	
CuSHL	2.0237 1.879	1.916 1.9093	1.3936 1.3483	1.2558 1.3094
CoSHL	1.8760 1.8958	1.9287 1.9027	1.3567 1.3452	1.2954 1.3011

## CONCLUSION

The Schiff base SHL was prepared successfully by condensation reaction between 4-Amino-3-mercapto-5-phenyl-4H-1,2,4-triazole and 2-hydroxy-1-naphthaldehyde, the tri-

azole Schiff base acts as a bidentate ligand through donor groups of phenolic and an imine and gave a good result compared with the theoretical calculation of bond length, especially (-C=N-), (-C-O-) bonds, CuSHL complex has longer bond length. The geometry of the prepared complexes was proposed the CoSHL complex has a tetrahedral geometry, NiSHL, and CuSHL complexes have a square planar geometry according to spectroscopy measurements. The valence electrons of metal ion affect physical properties and gave the semiconductivity behaviour to the free ligand. From the calculating energy gap using diffuse reflectance spectroscopy and theoretical study, CuSHL complex is more semiconductive, which indicates the electronic transitions from the HOMO to LUMO energy levels were easy, the tetrahedral CoSHL complex was less semi-conductive, due to difficult electronic transitions between the energy levels. As well as, the surface morphology of SHL and its complexes appearance more regular homogeneous distribution in NiSHL and CoSHL complexes.

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## DECLARATIONS

### Authors' contributions

All authors have equally contributed to this work.

### Conflict of interest

The authors declare that there is no conflict of interest.

### Ethical approvals

This work was conducted and approved by the Department of Chemistry, College of Science, Al-Nahrain University, Baghdad, Iraq.

### Data availability

The data associated with this work can be requested from the corresponding author.

### Funding resources

This work didn't receive any fund.

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