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RESEARCH ARTICLE

Optical and surface properties of Schiff base ligands and Cu(II) and Co(II) complexes

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Abstract

Objectives. To study the transition of electrons in 1,2-phenyl(4'-carboxy)benzylidene Schiff base ligand and transition metal ions, optical properties, as well as the surface chemistry of supported transition metals using diffuse reflectance spectroscopy (DRS); to study the roughness and morphology of the Schiff base ligand and its complexes using atomic force microscopy (AFM).

Methods. DRS, AFM, and Fourier-transform infrared spectroscopy instruments were used to identify electron transitions, optical properties, and surface morphology in Schiff base ligands and their complexes.

Results. The DRS revealed the d–d transitions and charge transfer shifts of all compounds, and helped identify the structure of the ligand. One of the optical properties studied was the energy gap calculation of the ligand and its complexes. The copper complex exhibited more semiconducting behavior with surface morphology properties such as surface roughness parameters lower than those of the ligand and the cobalt complex. This can be attributed to the smaller size of the copper atom, as well as lower electron transitions compared to the cobalt complex and the square planar bonding shape.

Conclusions. In Schiff base ligands, the reflectance spectrum bands reveal three electron transitions: $n\rightarrow\pi^*$, $\pi\rightarrow\pi^*$, and $\sigma\rightarrow\sigma^*$ transitions. In cobalt complexes, four transitions are indicated: $4A_2(F)\rightarrow 4T_1(F)$, $4A_2(F)\rightarrow 4T_1(P)$, charge transfer bands, and tetrahedral geometry. Copper complexes exhibit three transitions: $2B_1g\rightarrow 2A_1g$, $2B_1g\rightarrow 2Eg$, and charge transfer bands, with a square planar geometry for their structure. The energy gap calculations were 2.42, 2.29, and 2.30 eV, respectively. In the case of the SH ligands, copper complexes, and cobalt complexes, all compounds exhibited semiconductor properties. However, the complexes displayed increased conductivity due to the influence of the metal and coordination structure.

Keywords

energy gap, transition metal, morphology, roughness, reflectance, Schiff base ligand

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НАУЧНАЯ СТАТЬЯ

Оптические и поверхностные свойства лигандов на базе оснований Шиффа и комплексов Cu(II) и Co(II)

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Аннотация

Цели. Изучить переход электронов в 1,2-фенил(4’-карбокси)бензилиденовом лиганде основания Шиффа и в ионах переходных металлов, оптические свойства, а также химический состав поверхности нанесенных переходных металлов с помощью спектроскопии диффузного отражения (СДО); изучить шероховатость и морфологию лиганда на базе основания Шиффа и комплексов на его основе с использованием атомно-силовой микроскопии (АСМ).

Методы. Для идентификации электронных переходов, анализа оптических свойств и морфологии поверхности лигандов на базе оснований Шиффа и их комплексов были использованы СДО, АСМ и инфракрасная спектроскопия с преобразованием Фурье.

Результаты. СДО выявила d–d переходы и сдвиги в переносе заряда во всех соединениях и помогла идентифицировать структуру лиганда. Одним из изученных оптических свойств был расчет энергетической щели лиганда и его комплексов. Комплекс меди показал более ярко выраженные полупроводниковые свойства, в то время как морфологические свойства поверхности, такие как шероховатость, у комплекса меди были ниже, чем у лиганда и комплекса кобальта. Это можно объяснить меньшим размером атома меди, более низкими электронными переходами по сравнению с комплексом кобальта, а также плоскоквадратной геометрией.

Выводы. В лигандах на базе оснований Шиффа полосы спектра отражения обнаруживают три электронных перехода: $n\to\pi^*$, $\pi\to\pi^*$ и $\sigma\to\sigma^*$. В комплексах кобальта выявлены переходы 4A2(F)→4T1(F), 4A2(F)→4T1(P), полосы переноса заряда и тетраэдрическая геометрия. Комплексы меди демонстрируют переходы 2B1g→2A1g, 2B1g→2Eg и полосы переноса заряда, при этом структура комплексов меди имеет плоскоквадратную геометрию. Расчеты энергетического зазора составили 2.42, 2.29 и 2.30 эВ соответственно. В случае лигандов SH, комплексов меди и кобальта все соединения проявляли полупроводниковые свойства. Однако комплексы меди и кобальта проявляли повышенную проводимость из-за наличия металла и координационной структуры.

Ключевые слова

энергетическая щель, переходный металл, морфология, шероховатость, коэффициент отражения, лиганд на базе основания Шиффа

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INTRODUCTION

Understanding the chemical complexes of transition metal ions is crucial in identifying and quantifying their coordination. Diffuse reflectance spectroscopy (DRS) is the technique used to measure the light reflection of powdered samples in various regions of the ultraviolet, visible, and near-infrared (NIR) spectra. DRS is used to gather information about the outer-shell electrons of atoms and can be applied to detect both d–d and charge transfer transitions. DRS technology is suitable for on-site conditions. However, a disadvantage of DRS is the potential for band overlap, which can make the technique technically challenging. In a DRS spectrum, light scattered from the surface of a thick layer is measured as a function of wavelength [1].

Schiff base compounds, first developed by Hugo Schiff, are organic compounds. The functional group of these compounds is the imine group which forms through the condensation reaction of primary aromatic or aliphatic amines with carbonyl compounds (ketones or aldehydes) through nucleophilic addition [2]. Schiff bases have applications in a variety of fields, including biology, where they serve as ligands in inorganic and analytical chemistry [3]. They are an important class of coordination chemistry, involving the coordination between metal ions and azomethine nitrogen bonds [4].

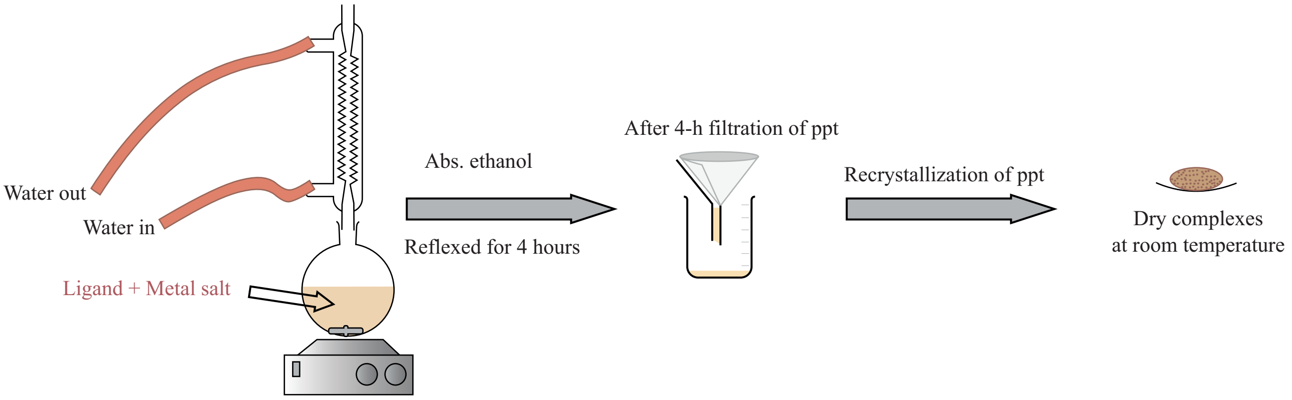
Due to their wide range of applications and unique electronic properties, Schiff base ligands are extensively studied in coordination chemistry. They have garnered significant attention for their roles in organic synthesis, metal refining, metallurgy, analytical chemistry, photography, and electroplating [5]. Moreover, they are extensively employed in materials science, playing roles in supramolecular chemistry, catalysts, photocatalysts, antioxidants, anticancer agents, antimicrobial agents, DNA binding, electrochemistry, and energy materials [6–20].

In this study, we investigated the surface morphology properties, including roughness and 3D surface images, of the Schiff base ligand and its complexes (copper and cobalt complexes). We also studied their optical properties, such as energy band gaps, indicating semiconductor properties, and their reflectance of light through DRS. The DRS data revealed d–d and charge transfer transitions of the metal ions.

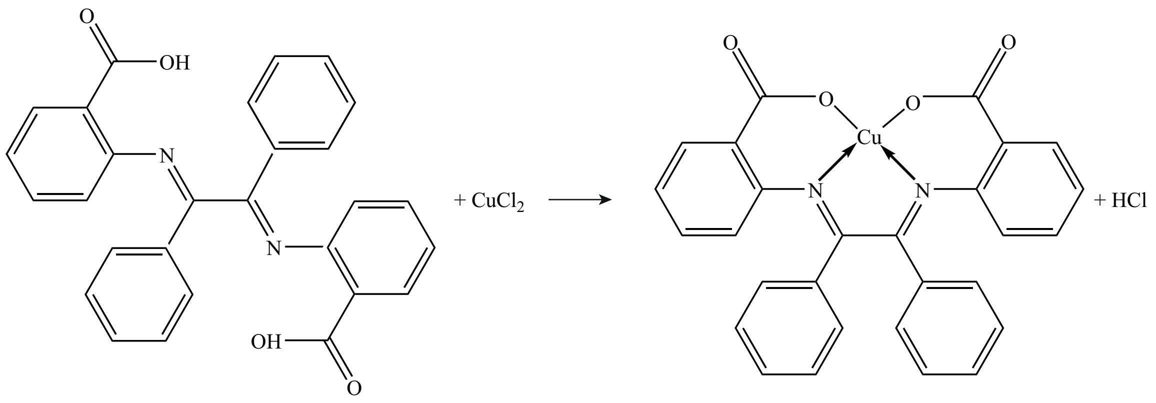
EXPERIMENTAL

1,2-phenyl(4’-carboxy)benzylidene Schiff base ligand and its cobalt complexes were prepared as previously reported, and their structures were confirmed [21]. The copper complex was also synthesized in a 1 : 1 molar ratio (metal : ligand) by adding 12.98 g (0.01 mol) of copper chloride anhydrous salt to a solution of 0.46 g (0.01 mol) of Schiff base ligand in 15 mL of absolute ethanol (Scheme 1). The mixture was then refluxed for 4 h. A dark brown precipitate of the Cu complex formed. It was then filtered, recrystallized with ethanol, and dried in an oven at 60°C. Attenuated total reflection infrared (ATR-IR) spectroscopy was performed, in order to identify the complex (Scheme 2).

The following instruments of diffuse reflectance spectroscopy (DRS) were used for identifying electron transitions, optical properties, and surface morphology in Schiff base ligands and their complexes: (AvaLight-DH-S-BAL-2048 UV–Vis, *Avantes*, Netherlands) in a wavelength range of 230–1100 nm, atomic force microscope (AFM) (AA2000, *Angstrom Advanced Inc.*, USA, contact mode, atmospheric conditions), and Fourier-transform infrared spectrometer (FTIR) (*Bruker ALPHA*, USA) in the transmission range 400 to 4000 cm^{-1}.



Scheme 1. Steps of copper complex preparation



Ligand

Copper chloride anhydrous

Scheme 2. Structure of copper complex

RESULT AND DISCUSSION

IR analysis

The ATR-IR spectra of both the ligand and the cobalt complex were identified as previously reported [21]. In the case of the copper complex, it was observed that the complex formed with the following linkage: Cu (ligand) [22–25]. Complications arose between the copper metal and the nitrogen atom in the imine, as well as the oxygen atom in the hydroxyl group. This resulted in the disappearance of certain bands and shifts in others. Specifically, the imine band shifted from 1583 cm^{-1} in the ligands to 1533 cm^{-1} in the Cu complexes. Additionally, there was a shift in the carbonyl band from 1661 cm^{-1} in the ligand to 1799 cm^{-1} in the Cu complexes. Table 1 displays some important IR spectra bands of the SH ligand and CuL_2 complex, affected and shifted as a result of the complex formation.

Table 1. IR spectra bands (cm^{-1}) of SH ligand and CuL_2 complex

Functional groups	SH ligand bands, cm^{-1}	CuL_2 complexes bands, cm^{-1}
ν O–H	3320	–
ν C–H Ar	3066	3063
ν C=O	1661	1799
ν C=N	1583	1533
ν C–O	1451	1433

Optical properties

The photophysical properties of the SH ligand, copper complexes, and cobalt complexes were measured using DRS (AvaLight-DH-S-BAL-2048 UV–Vis) in the wavelength range of 238–1000 nm, covering the ultraviolet, visible, and NIR regions. DRS spectroscopy is a valuable analytical tool for obtaining information about outer-shell electrons. It is particularly useful in determining d–d transition and charge transfer spectra in inorganic complexes [1]. Figure 1 depicts the reflectance of light from the surface, while Table 2 presents the DRS data for the ligand, copper complex, and cobalt complex in terms of wavelength and wavenumber. In the Schiff base ligand, the reflectance spectrum shows absorption wave numbers at 11682.24, 13986.01, 20449.90, and 26455.03 cm^{-1}, respectively. These wave numbers may be assigned to $n\rightarrow\pi^*$, $\pi\rightarrow\pi^*$, and $\sigma\rightarrow\sigma^*$ transitions. The positions of these bands can be utilized, in order to calculate shifts in the corresponding positions in the spectra of the metal complexes [26, 27].

The reflectance spectrum of the cobalt complexes displays absorption wave numbers at 10810.81, 17574.69, 26455.03, and 37593.98 cm^{-1}, respectively. They can be attributed to transitions $4A_2(F)\rightarrow 4T_1(F)$, $4A_2(F)\rightarrow 4T_1(P)$, and charge transfer bands. Active 1: Researchers have assigned several transitions in the cobalt(II) complexes to the tetrahedral geometry of cobalt complexes, with a coordination number of four [28–30].

Absorption wave numbers at 37735.85, 26455.03, and 14771.05 cm^{-1} were observed in the reflectance spectrum of the copper complexes. The various transitions in the copper(II) complexes indicate a square planar structure geometry with a coordination number of four [31–34].

In addition, reflectance describes the amount of light reflected from a surface. As shown in Fig. 2, the surface morphology and structure of the compound affect the reflectance. The ligand reflects more light compared to its complexes, indicating higher reflectance and some absorption. The reflectance of the ligand is higher when compared to its complexes, indicating that more light is reflected and some is absorbed. In the cobalt complex, a higher percentage of light is absorbed in the visible range, while more light is reflected in the NIR range, possibly due to the transition $4A_2(F)\rightarrow 4T_1(F)$.

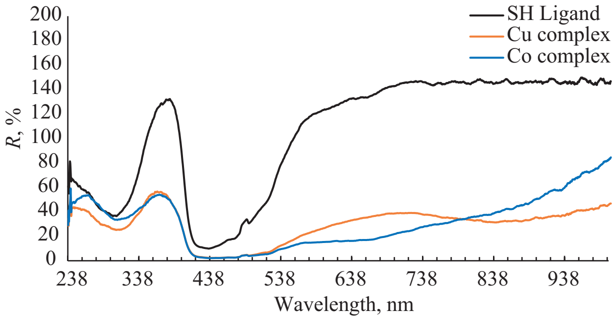


Fig. 1. DRS spectra of SH ligand, copper complex, and cobalt complex

Another optical property which was studied involved determining the energy gap. This was calculated using the DRS data through the application of the Kubelka–Munk theory [35, 36].

$$F(R_\infty) = (1 - R_\infty)^2/2R_\infty, \quad (1)$$

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

$$\alpha = F(R_\infty),$$

$$[F(R_\infty)\, h\nu]^2 = C_2(h\nu - E_g), \quad (2)$$

where F is the Kubelka–Munk function; R_∞ is the sample reflection coefficient; α is the absorption coefficient; E_g is the energy gap; $h\nu$ (photon energy) = $1240/\lambda$; h is the Plank constant; λ is the absorption wavelength; C_1 is a frequency-independent proportionality constant that includes a matrix element of transition from the valence band to the conduction band; $C_2 = C_1^2$.

Figure 2 shows the plots of $[F(R_\infty)\cdot h\nu]^2$ against photon energy ($h\nu$) for the SH ligand, copper complexes, and cobalt complexes. The intersection point on these plots corresponds to the band gap values. The energy gap calculations were 2.42, 2.29, and 2.30 eV for the SH ligand, copper complexes, and cobalt complexes, respectively. All of these compounds exhibited semiconductor properties, but the complexes displayed higher conductivity due to the influence of the metal and coordination structure.

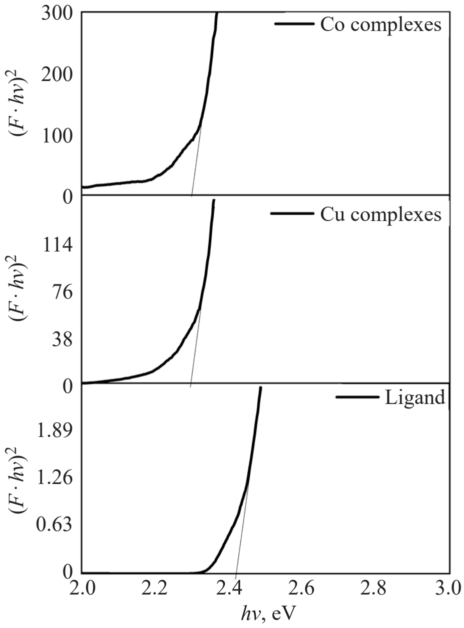


Fig. 2. Energy gap of compounds

Surface morphology study

Surface morphology properties were assessed using AFM. The surface morphology of the Schiff base ligand and its complexes (copper and cobalt) was examined by analyzing 2D and 3D AFM images, changes in surface roughness, cross-sectional profiles, and particle diameters.

Figure 3 presents both 2D and 3D AFM images. The ligand exhibited a rough surface, likely due to its inherent structural features and length, which manifested as contiguous clusters on the surface. In the copper complex, the surface distribution appeared nonuniform.

Table 2. DRS data of ligand, copper complex, and cobalt complex

Ligand		Copper complex		Cobalt complex	
Wavelength, nm	Wavenumber, cm^{-1}	Wavelength, nm	Wavenumber, cm^{-1}	Wavelength, nm	Wavenumber, cm^{-1}
378	26455.03	265	37735.85	266	37593.98
489	20449.90	378	26455.03	378	26455.03
715	13986.01	677	14771.05	569	17574.69
856	11682.24	–	–	925	10810.81

This was possibly due to the stereochemistry of the structure and structural rotations of the complex, as a result of which certain areas appeared as simple particle assemblies. Conversely, in the cobalt complex, the surface was rougher and exhibited heterogeneous distribution, which can likely be attributed to the larger size of cobalt atoms when compared to copper.

Figure 4 indicates the grain distribution on the surface and the histogram of particle diameters. The average diameter was 87.91, 93.87, and 86.02 nm for the ligand, Cu complex, and Co complex, respectively.

Figure 5 also presents cross-sections which reveal both smooth and rough surfaces. In the ligand, the rough surface is evident as a wider peak on the chart, whereas

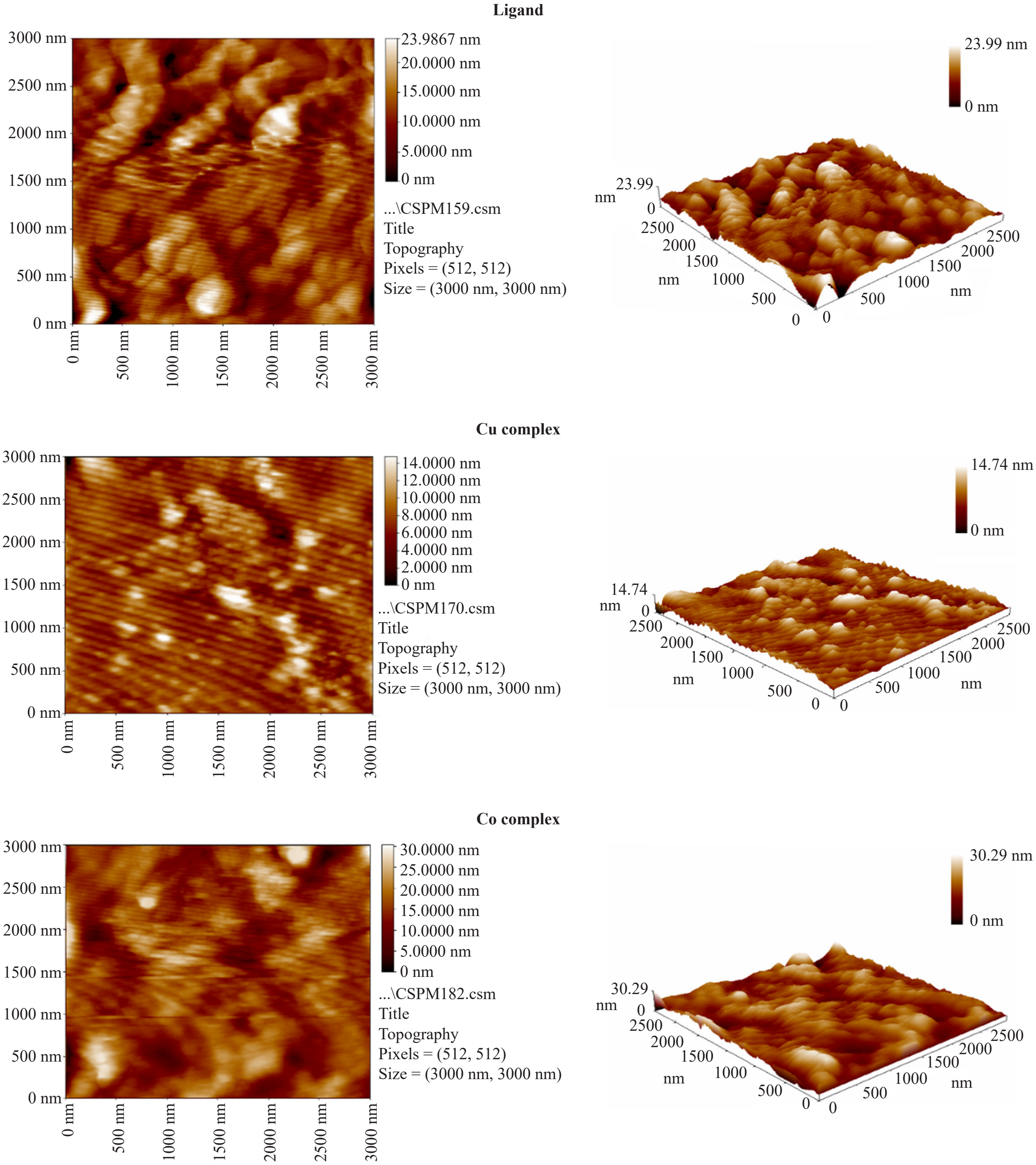


Fig. 3. 2D and 3D AFM image

in copper, the surface is less rough, resembling a sharper peak. In contrast, the cobalt complex exhibits a rougher surface when compared to the ligand, resulting in a wider peak width. This can be attributed to the tetrahedral coordination of the cobalt complex, affecting the surface characteristics.

Values for the surface roughness are provided in Table 2. Here it can be seen that the Cu complex has lower roughness compared to both the ligand and the Co complex.

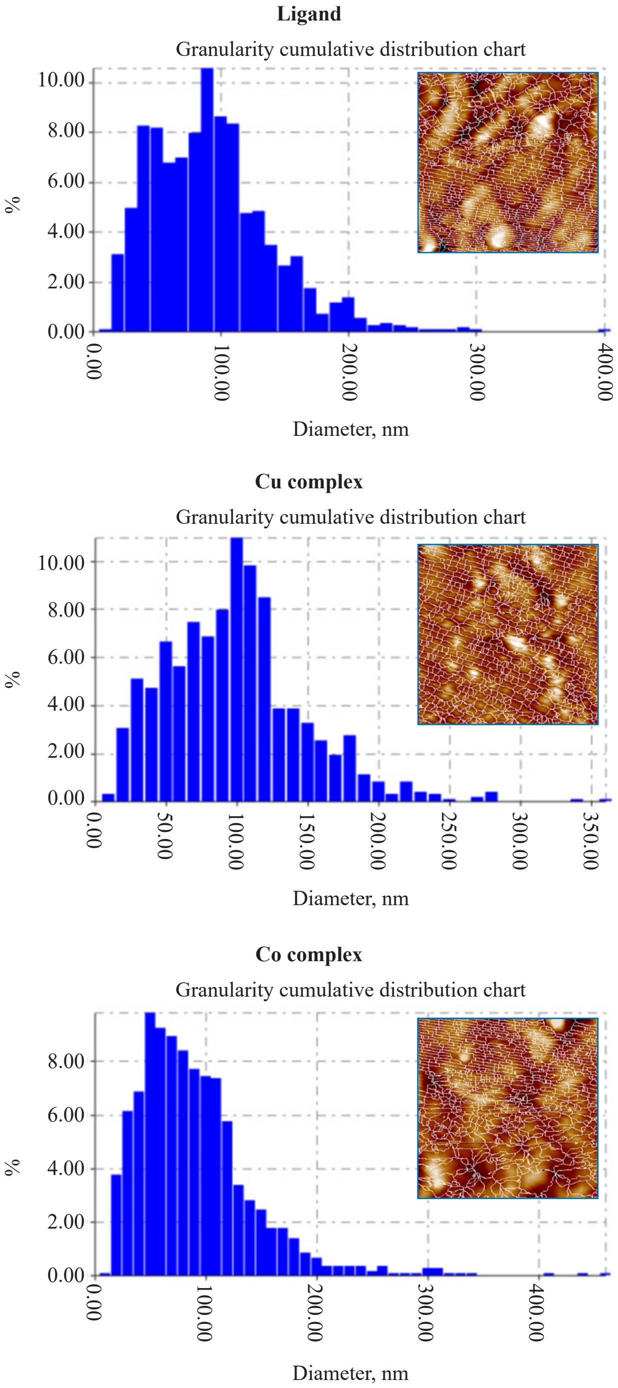


Fig. 4. Grain and histogram distribution of particle surface

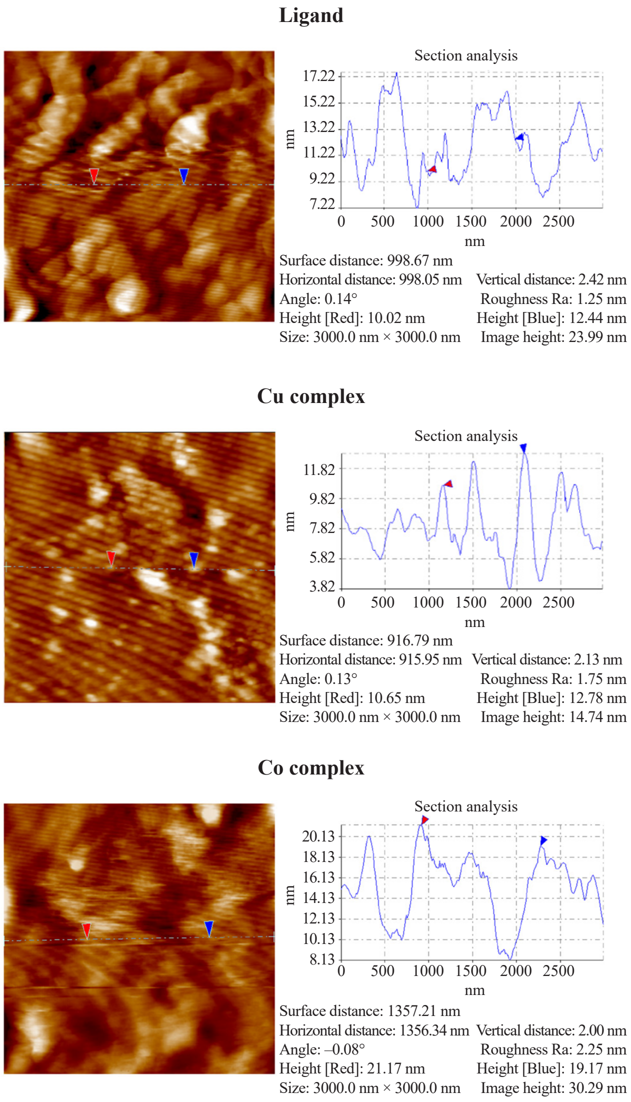


Fig. 5. Cross section of surface

Table 3. Roughness parameter data

Roughness parameter	Ligand	Cu complex	Co complex
Average rough	3.07	1.43	3.31
RMS	3.92	1.92	4.24
Peak peaking	24	14.7	30.1

CONCLUSIONS

The optical and surface properties of the Schiff base ligand complex are influenced by the choice of metal. Cobalt is more significantly affected due to its tetrahedral coordination structure. This results in four transition electrons shifting from the ground state to the excited state. In contrast, the copper complex forms a square planar coordination structure with only three transition electrons, impacting the energy gap and making copper more semiconductor-like than the cobalt complex.

Furthermore, the surface of the copper complex exhibits lower roughness parameters when compared to the cobalt complex, which due to its tetrahedral structure and the larger size of cobalt atoms has higher values.

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Authors' contributions

Alaa Adnan Rashad, **Dina A. Najeeb**—experimental work, writing original draft, and editing.

Shaymaa M. Mahmoud—correction of the research concept.

Evon Akram—research concept, processing experimental data.

Khalid Zainulabdeen, **S. Dulaimi,** and **Rahimi M. Yuso**—processing experimental data and checking English language.

The authors declare no conflicts of interest.

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