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Synthesis and Characterization of N_2O_2 Type Schiff Base Ligand with Salicylaldehyde Derivate and Its Metal Complexes

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Abstract: Schiff bases and their metal complexes, which play a role in various biological processes, are one of the most important classes of organic and inorganic compounds. Due to their high use in the field of health, it is important to introduce these compounds into the literature through synthesis and research studies. In this study a new Schiff base ligand was synthesized by the reaction of 4-diethylamino-2-hyrdoxybenzaldehyde with o-aminophenol. This ligand was used to prepare Co(II), Ni(II), Cu(II) and Zn(II) complexes. The structural characterization of the synthesized ligand and metal complexes was elucidated by various spectroscopic and thermal analysis methods such as FT-IR, ^{1}H-NMR, ^{13}C-NMR, UV-Vis., XRD, SEM and TGA. As a result of these characterization studies, it was determined that the synthesized Schiff base acted as an N_2O_2 type tetradentate ligand and that the metal ions were bound to the ligand via phenolic oxygen and azomethine nitrogen.

Keywords: Imine, Ligand, Metal Complex, Schiff Base, Structural Characterization.

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1. INTRODUCTION

Schiff bases or azomethines, which contain an imine group (-HC=N-) in their structure, are among the most preferred compounds today due to their biological activities. They are synthesized by the reaction with a compound containing carbonyl group and an active amine under appropriate conditions, are among the widely used ligands owing to structural, biological, and pharmacological properties (1-3). These are precious ligands due to their ability to coordinate different metals and play a role in the formation of metal complexes due to the coordinator feature of the nitrogen atom in their structure. Metal complex formation from the ligands is important for effective and specific catalysts for oxidation, reduction, hydrolysis, and other transformations of organic and inorganic chemistry (4, 5). In addition, they are in the class of compounds that have been studied extensively due to their flexibility and selectivity properties.

Schiff base ligands and complexes are increasingly used in various fields such as medicine and pharmacy, preparation of some drugs, analytical chemistry, biological systems, agriculture, dyestuff production, cosmetics, polymer production, plastic industry, electronics industry, aircraft industry and liquid crystal technology (6, 7). In recent studies, it has been determined that they have a lot of biological and pharmacological activities such as antibacterial, antimicrobial, anti-inflammatory, anti-HIV, antioxidant, antitumor, anticancer, antiulcer, diuretic, and antipyretic activities (8-10).

2. EXPERIMENTAL SECTION

2.1. Materials and Instruments

In the synthesis of ligand and metal complexes, 4-diethylamino-2-hyrdoxybenzaldehyde (98%, Sigma-Aldrich), *o*-aminophenol (99%, Sigma-Aldrich), cobalt(II) acetate tetrahydrate (99%, Merck), nickel(II) acetate tetrahydrate (%99.9, Sigma-Aldrich), copper(II) acetate monohydrate (99%, Merck), zinc(II) acetate dihydrate (99.5%, Merck), *p*-toluenesulfonic acid (98%, Merck) as precursors and acetone, ethanol, methanol, diethyl ether, dimethyl sulfoxide as solvents at analytical reagent grade were used.

The infrared spectrum of the synthesized compounds was taken with Thermo Scientific Nicolet 6700 FT-IR

Spectrophotometer and their thermogravimetric analyses were performed using the Shimadzu model TGA-50 device. Elemental analyses were taken with LECO-932 CHNSO model elemental analyzer. ^{1}H-NMR and ^{13}C-NMR spectra were taken on Bruker DPX-400, 400 MHz high performance digital NMR spectrometer. UV spectra were recorded with Shimadzu 1240 model UV-Vis Spectrophotometer. Magnetic susceptibilities were measured by a modified Gouy method. In addition, the FEI QUANTA FEG 450 model Scanning Electron Microscope (SEM) the Panalytical was used to determine the morphology of ligand, and Empyrean X-Ray Diffraction (XRD) Device (using $CuK\alpha 1$ $\lambda=1.5405$Å radiation with 2θ of 10–100°, operated at 45 kV and 40 mA) was used for XRD patterns of the ligand.

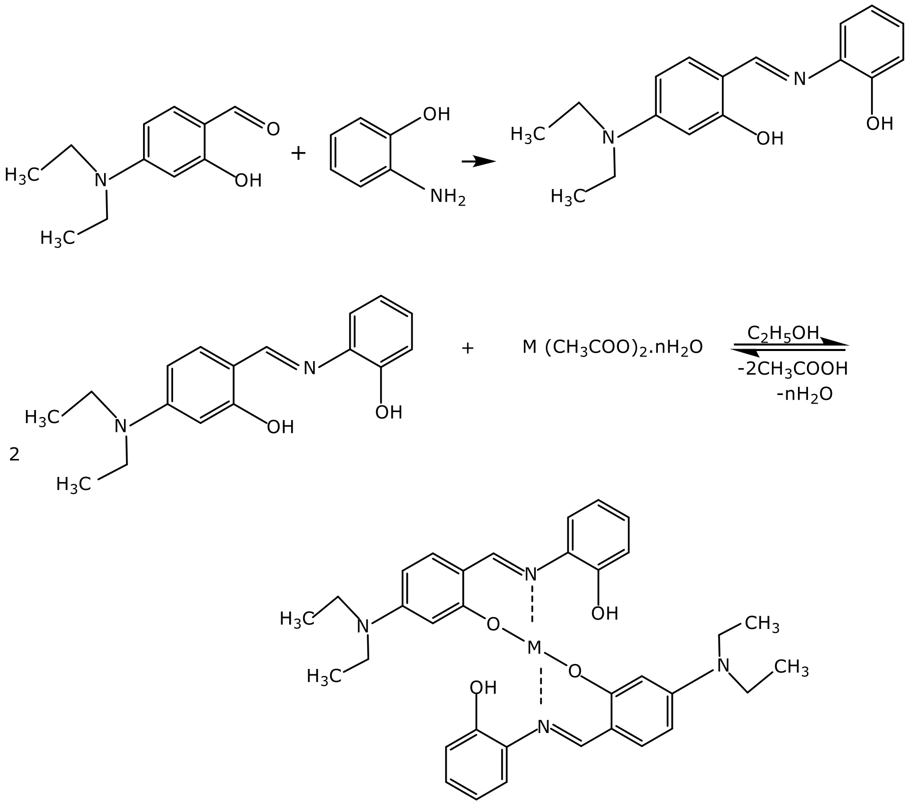
2.2. Synthesis of Compounds

2.2.1. Synthesis of Ligand

4-Diethylamino-2-hyrdoxybenzaldehyde (1.93 g, 10 mmol) in 20 mL hot ethyl alcohol was dripped to 20 mL ethyl alcohol solution of *o*-aminophenol (1.09 g, 10 mmol) and (0.01 g) *p*-toluenesulfonic acid. The solution was refluxed for 4 h. The dark yellow compound obtained as a result of the reaction (Figure 1) was filtered off, washed several times with hot distilled water and diethyl ether, recrystallized from ethanol, and dried at room temperature (11, 12). FT-IR (v cm^{-1}, KBr); 3430 (phenolic, O-H), 3050 (aromatic, C-H), 2975-2930 (aliphatic, C-H), 1618 (-C=N), 1568-1435 (C=C) and 1287 (C-O). ^{1}H-NMR (δ, DMSO-d$_6$); 14.20 (s, 1H), 9.62 (s, 1H), 8.64 (s, 1H), 7.29 (d, 1H), 7.04 (s, 1H), 7.00 (s, 1H), 6.91 (s, 1H), 6.84 (d, 1H), 6.28 (s, 1H), 5.98 (s, 1H), 2.52 (q, 4H), 1.27 (t, 6H). ^{13}C NMR (DMSO-d$_6$); δ 166.19 (-C=N), 159.14 (s), 152.10 (s), 150.56 (s), 135.11 (s), 134.43 (s), 126.64 (s), 120.05 (Aromatic-C), 119.01 (s), 116.65 (s), 109.50 (s), 104.14 (s), 97.69 (s), 44.35 (-CH$_2$), 13.08 (-CH$_3$).

2.2.2. Synthesis of metal complexes

The ligand (0.57 g, 1.00 mmol) was dissolved in 20 mL ethyl alcohol and then metal salts [$Co(CH_3COO)_2.4H_2O$ (0.125 g, 0.50 mmol), $Ni(CH_3COO)_2.4H_2O$ (0.124 g, 0.50 mmol), $Cu(CH_3COO)_2.H_2O$ (0.10 g, 0.50 mmol), $Zn(CH_3COO)_2.2H_2O$ (0.110 g, 0.50 mmol)] in 20 mL ethyl alcohol were dripped to ligand solutions separately. These solutions were heated under reflux for 2-4 h (Figure 1). The compounds were filtered off, washed several times using hot distilled water and diethyl ether, and dried at room temperature (13, 14).



M: Co^{+2}, Ni^{+2}, Cu^{+2}, Zn^{+2}

Figure 1: Synthesis of 2-[(4-(diethylamino)-2-hydroxybenzilidene)amino)]phenol and metal complexes (C$_{1-4}$).

3. RESULTS AND DISCUSSION

3.1. Structural Characterization

The elemental, spectral, and thermal analyses were used to investigate the structure of the synthesized compounds (15, 16). No mass loss was observed in the thermograms of the synthesized compounds from 25 °C to 270 °C. This means that there was no crystal water at the compounds (17, 18). As seen in Table 1, elemental analysis showed that the ligand:metal ratio was 2:1 at the compounds. That is, the data obtained from the thermogram supports the results of the elemental analysis.

Table 1: Some analytical and physical data of ligand and metal complexes.

Compound	Formula	W (g/mol)	Color	m.p (°C)	μ_{eff}	Yield (%)	Elemental Analysis, % Calculated (Found)		
							C	H	N
Ligand	$C_{17}H_{20}N_2O_2$	284.359	Dark Yellow	132-134	-	84.00	71.81 (71.79)	7.09 (7.04)	9.85 (9.78)
[Co(L)$_2$]	$CoC_{34}H_{38}N_4O_4$	625.635	Dark Brown	>300	4.09	68.00	65.27 (65.19)	6.12 (6.07)	8.95 (8.91)
[Ni(L)$_2$]	$NiC_{34}H_{38}N_4O_4$	625.395	Light Brown	>300	2.68	62.00	65.30 (65.28)	6.12 (6.06)	8.96 (8.81)
[Cu(L)$_2$]	$CuC_{34}H_{38}N_4O_4$	630.248	Dark Green	>300	1.73	66.00	64.80 (64.76)	6.08 (6.02)	8.89 (8.78)
[Zn(L)$_2$]	$ZnC_{34}H_{38}N_4O_4$	632.092	Dark Yellow	>300	Dia.	69.00	64.61 (64.59)	6.06 (6.01)	8.86 (8.79)

As seen in Figure 2, a wide band was observed at 3430 cm^{-1} assignable to the phenolic O-H group. Characteristic band of imine group (-CH=N-) was observed at 1618 cm^{-1} as a sharp peak. The other bands at 3050 cm^{-1}, 2975-2930 cm^{-1}, 1568-1435 cm^{-1}, and 1287 cm^{-1} were assigned to the aromatic C-H, the aliphatic C-H, the C=C stretching vibration, and the phenolic C-O group, respectively (19, 20). In addition, the absence of the –C=O stretching vibration of 4-diethylamino-2-hydroxybenzaldehyde at 1637 cm^{-1} and the -NH$_2$ stretching vibration of aromatic amine at 3440 cm^{-1} in the spectrum showed that the reaction was completed. Instead of the two bands, -CH=N- stretching vibration was formed.

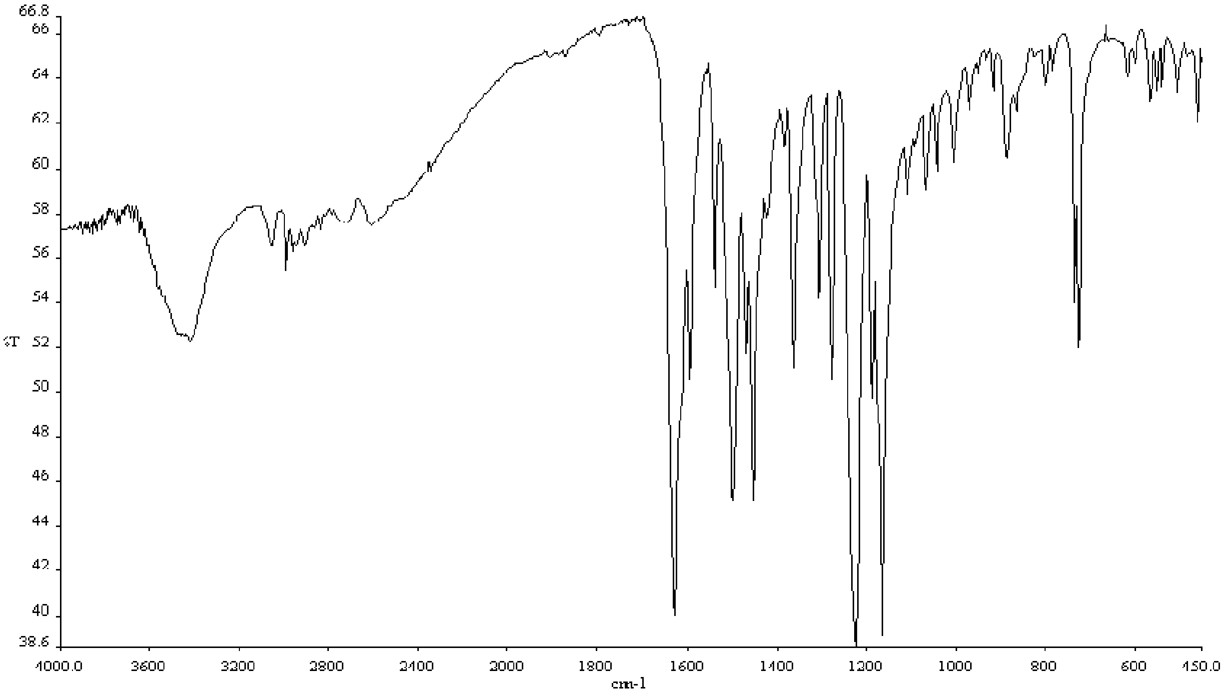
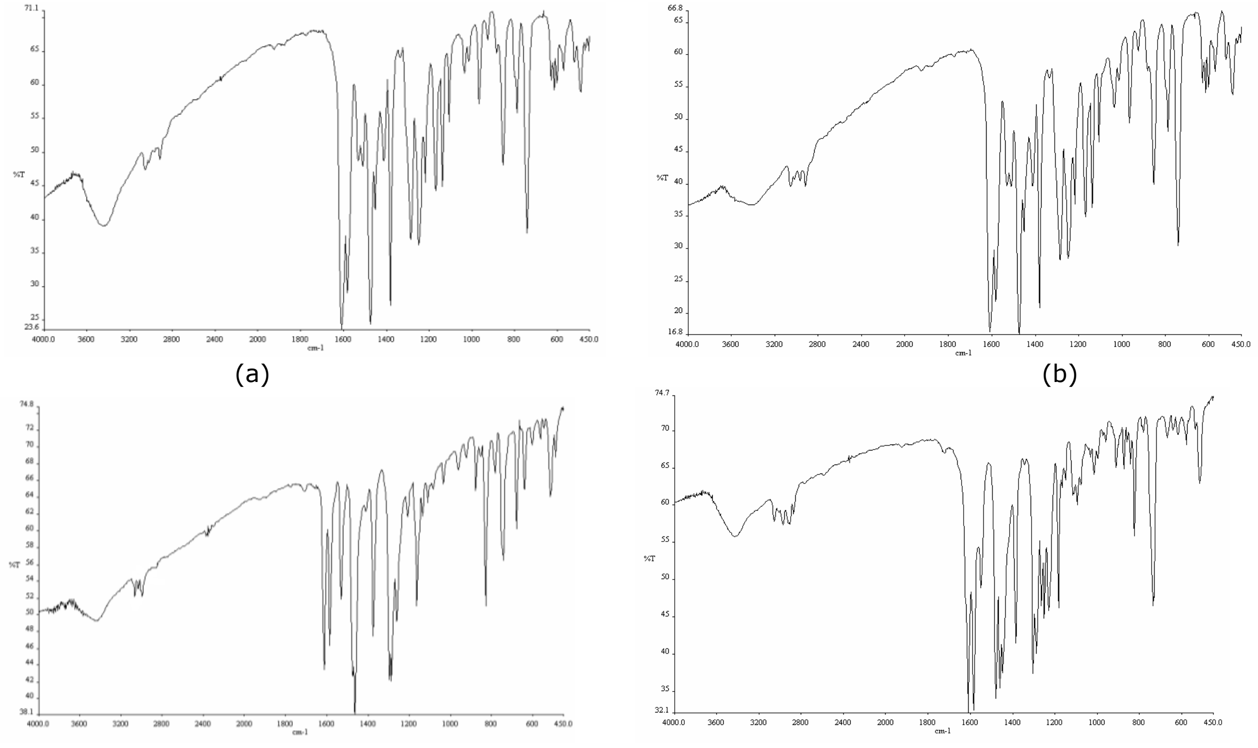


Figure 2: Infrared spectrum of ligand.

The -CH=N- stretching vibration of ligand, which was observed at 1618 cm^{-1}, showing the existence of imine group, shifted to the low-frequency region to 1602-1609 cm^{-1} in metal complexes (Figure 3). The observed shift indicates that N atom at the imine group plays a role in the formation of M-N bond. The nitrogen atom donated its unpaired electrons to metal ions (21, 22). In addition, the shifts in the peak observed at 1287 cm^{-1} support that deprotonated oxygen forms coordinated bonds with metal ions in complex formation.

The ^{1}H-NMR spectrum showed that the integral ratios were consistent with the expected number of protons in each group (23, 24). At the spectrum of the ligand (Figure 4), a proton singlet was observed at 14.20 ppm, which regard to the phenolic OH group. The other singlet was observed at 9.62 ppm belongs to the proton of the –OH group which is adjacent to diethylamino group. The chemical shift of H proton of the imine group at ligand was observed as a singlet at 8.64 ppm. Protons belonging to aromatic ring were observed as multiples in the range of 7.29-5.98 ppm. While the chemical shift of the H proton of –CH$_2$ group was observed at 2.52 ppm, the chemical shift of group –CH$_3$ was observed at 1.27 ppm.



(a) (b) (c) (d)

Figure 3: Infrared specta of metal complexes [(a) $[Co(L)_2]$, (b) $[Ni(L)_2]$, (c) $[Cu(L)_2]$, (d) $[Zn(L)_2]$].

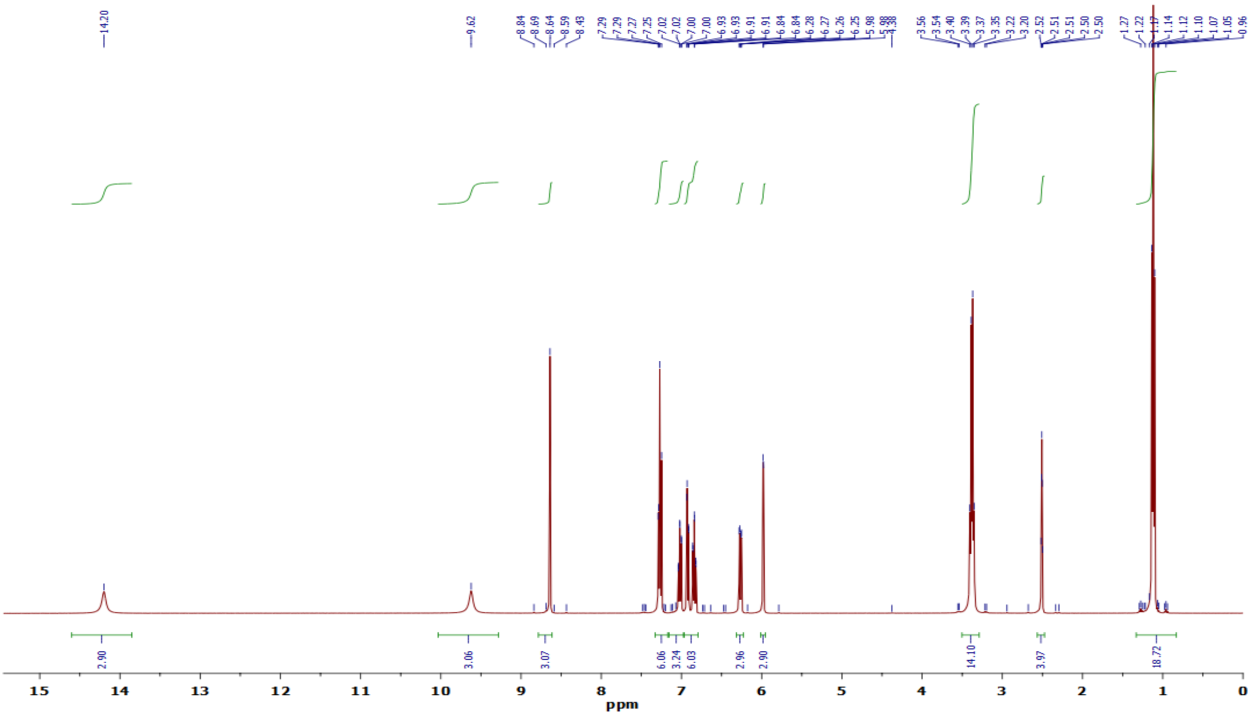


Figure 4: ^{1}H-NMR spectrum of ligand.

In the ^{13}C-NMR spectrum of ligand (Figure 5), chemical shift of the C atom of the –OH group which is adjacent to the diethylamino group, was observed at 152.10 ppm. The chemical shift of carbon atom of the other –OH group was observed at 150.56 ppm (25, 26). The chemical shift observed at 166.19 ppm belongs to the imine group carbon atom. The chemical shift of C-N group C was observed at 159.14 ppm and C atoms of the aromatic ring were observed in the range of 135.11-97.69 ppm. The chemical shifts of the C proton of $–CH_2$ group and $–CH_3$ group were observed at 44.35 ppm and 13.08 ppm, respectively (27).

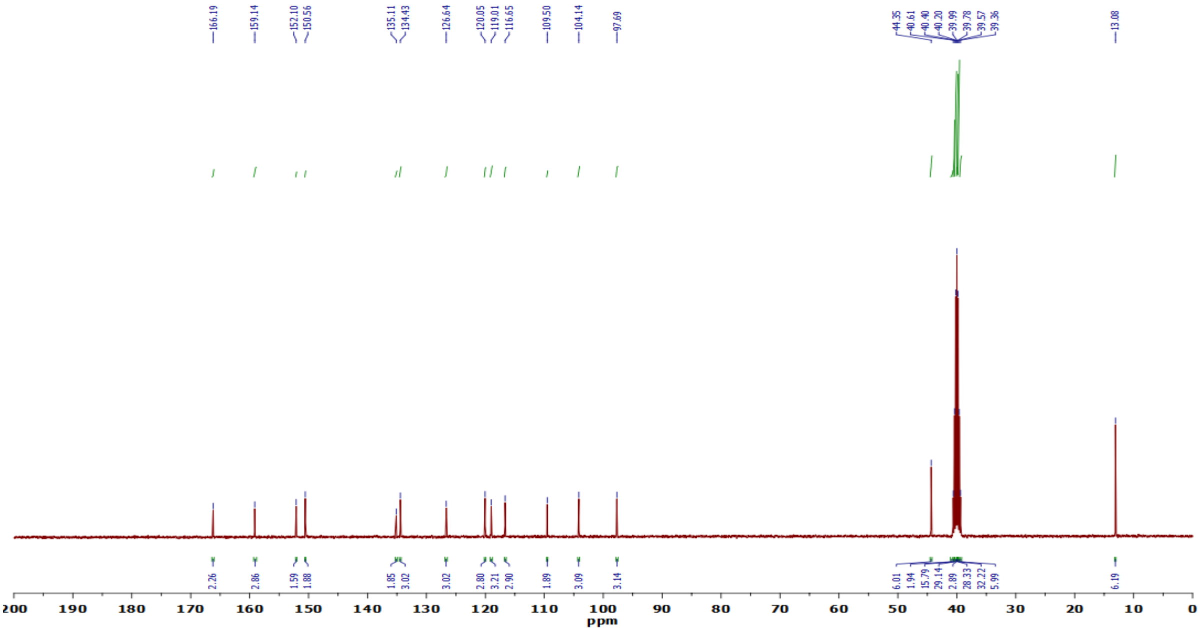


Figure 5: ^{13}C-NMR spectrum of ligand.

The structural characterization of the metal complexes was supported by magnetic susceptibility (μ_{eff}) values. μ_{eff} of Co(II) complex was 4.09 B.M., corresponding to 3 electrons and μ_{eff} of Ni(II) complex was 2.68 B.M., corresponding to 2 electrons. For Cu(II) complex, μ_{eff} value was 1.73 B.M., corresponding to 1 electron. These paramagnetic complexes preferred tetrahedral geometry and the diamagnetic Zn(II) complex preferred tetrahedral structure because of d_{10} configurations (28, 29).

Detailed information about the electronic structures of the compounds can be obtained from UV-Vis. spectra. UV-Vis. measurements of compounds were taken with ethanol. The absorption band of the ligand was at 332 nm involving the $\pi \rightarrow \pi^*$ electronic transition (30, 31). The $n \rightarrow \pi^*$ transition of the unpaired electrons of the nitrogen atoms of the imine group was observed as a broad peak at 412 nm (Figure 6). Considering the peak intensities, a decrease was observed in the peak intensities of the metal complexes compared to the ligand. Otherwise, the band of the ligand obtained from $n \rightarrow \pi^*$ shifted to 390-415 nm at the complexes. This caused a wavelength shift during the formation of the $n \rightarrow \pi^*$ transition complexes of unpaired electrons of the nitrogen atom of imine group. This shift showed interaction between -CH=N- group and metal ions. The absorption bands appearing in the 425-450 nm range at the complexes indicated charge transfer transitions.

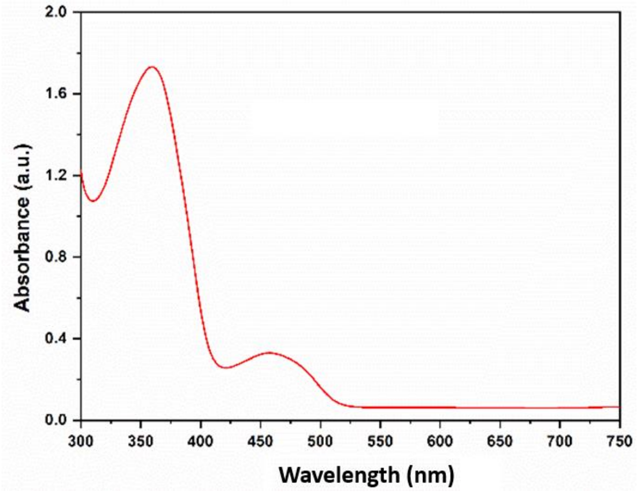


Figure 6: UV-Vis. spectrum of ligand.

The surface morphology of the ligand was examined with Scanning Electron Microscope analysis (Figure 7). As seen in Figure 6, surface morphology of the ligand was determined to contain compact, dense nanoparticles and localized clusters (32, 33).

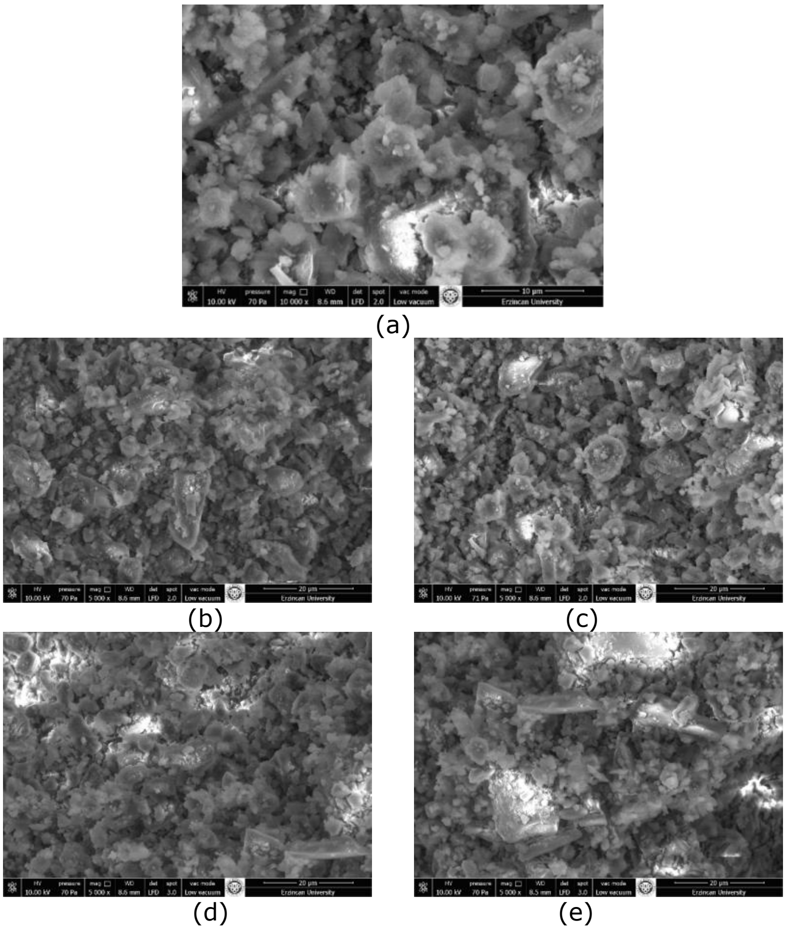


Figure 7: SEM image of ligand (a) and metal complexes [(b) $[Co(L)_2]$, (c) $[Ni(L)_2]$, (d) $[Cu(L)_2]$, (e) $[Zn(L)_2]$].

The structural analysis of the ligand was carried out by using XRD varying the diffraction angle, 2θ from 10 to 100° (Figure 8). As seen in XRD pattern, the ligand has sharp crystalline peaks. Also, XRD analysis showed the presence of two important characteristic peaks at 2θ=18.54° and 2θ=21.28° of imine group of the ligand (34, 35).

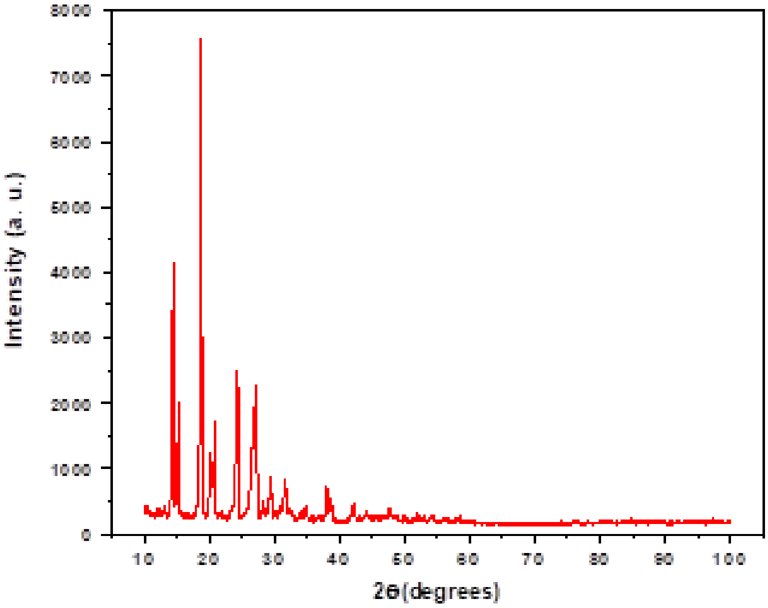


Figure 8: XRD pattern of ligand.

4. CONCLUSION

N_2O_2 type ligand, 2-[(4-(diethylamino)-2-hydroxybenzilidene)amino)]phenol, was synthesized from the reaction of an aromatic aldehyde with *o*-aminophenol. The four metal complexes were synthesized from the reaction with metal acetates and synthesized ligand. The characterization studies were completed by using instrumental analysis methods such as FT-IR, ^{1}H-NMR, ^{13}C-NMR, UV-Vis, XRD, SEM, TGA, and magnetic susceptibility. It was determined that in all complexes, the ligand acted as a bidentate chelate by binding to metal ion through imine nitrogen and phenolic oxygen, and M:L ratio for all complexes was 1:2 and the ligand existed as a tetradentate ligand.

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