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New Mixed Ni(II), Cu(II), and Co(II) Complexes derived from 2-((2-mercaptophenylimino)methyl)-4-methoxyphenol and 1,10-Phenanthroline: Synthesis and Characterization

2-((2-merkaptofenilimino)metil)-4-metoksifenol ve 1,10-fenantrolinden Türetilen Yeni Karışık Ni(II), Cu(II) ve Co(II) Kompleksleri: Sentez ve Karakterizasyon

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Abstract

Three new mixed Ni(II), Co(II) and Cu(II) complexes have been created using 2-((2-mercaptophenylimino)methyl)-4-methoxyphenol and 1,10-phenanthroline. FT-IR, Elemental analysis, magnetic susceptibility, UV-Vis and molar conductivity technique have all been used to explore the structural characterisation of these compounds. Magnetic susceptibility readings for the L-Cu, L-Ni, and L-Co complexes were 1.97 BM, 2.92 BM, and 2.84 BM, respectively. The L-Co complex is thought to be antiferromagnetic. The structures of the mixed ligand metal complexes are thought to be octahedral geometry. There are no conductivity properties in the complexes; however, they exhibit molar conductivities that range from 2.46 to 9.04 µS/cm.

Keywords: Schiff base, Mixed Ligand Metal complex, 1,10-phenantroline, Characterization

Öz

2-((2-merkaptofenilimino)metil)-4-metoksifenol ve 1,10-fenantrolinden üç yeni karışık Ni(II), Co(II) ve Cu(II) kompleksi sentezlenmiştir. Bu bileşiklerin yapısal karakterizasyonu, FT-IR, elementel analiz, manyetik duyarlılık, UV-Vis ve molar iletkenlik teknikleri kullanılarak araştırılmıştır. Manyetik duyarlılık ölçümleri L-Cu, L-Ni ve L-Co kompleksleri için sırasıyla 1.97 BM, 2.92 BM, ve 2.84 BM olarak kaydedilmiştir. L-Co kompleksinin antiferromanyetik özellikte olduğu düşünülmektedir. Karışık ligand metal komplekslerinin yapılarının oktahedral geometriye sahip olduğu düşünülmektedir. Komplekslerin molar iletkenlik değerleri 2.46-9.04 µS/cm arasında olduğundan dolayı iletkenlik göstermemektedir.

Anahtar kelimeler*: Karışık Ligand Metal kompleksi, Schiff baz, 1,10-fenantrolin, Karakterizasyon*

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1. Introduction

The condensation of amines and carbonyl compounds, in which the carbonyl group is converted to an azomethine group or an imine group, results in the formation of Schiff bases, yet another type of adaptable chemical molecule. (Schiff, 1864; Fabbrizzi, 2020; Surendar et al., 2021; Surendar et al., 2021b). Schiff bases can also be utilized as a versatile ligand in the formation of metal coordinated complexes (Al Zoubi et al., 2017; Chohan et al., 1997). In coordination chemistry, the design and development of Schiff bases with many donor sites is a topic that is continually changing (Sahraei et al., 2017a; Sahraei et al., 2017b). In order to create transition metal complexes with structural, magnetic, electrical, and pharmacological characteristics, a sizable library of Schiff bases is frequently used (Bhardwaj et al., 2012; Ibrahim et al., 2021; Jafari et al., 2017). Schiff's bases have produced a huge number of medicinally active molecules (Kajal et al., 2013; da Silva et al., 2011). They have been discovered to have good anti-inflammatory, analgesic, and anti-tubercular qualities, among other things (Sondhi et al., 2006; Miri et al., 2013).

Here in we present, the preparation and structural elucidation of ligand and metal complexes. Molar conductivity experiments confirmed the complexes' electrolytic behavior. The interaction of 2-((2-mercaptophenylimino)methyl)-4-methoxyphenol and 1,10-phenanthroline produced the mixed ligand metal complexes. Mixed ligand metal complexes were investigated using a variety of techniques, including UV-Vis, elemental analysis, FT-IR, magnetic susceptibility, and molar conductivity.

2. Experimental

2.1. Material and method

Without any purification, commercial solvents and compounds from Sigma-Aldrich were utilized in the investigation. Table 1 includes a list of the device manufacturers and models utilized in the investigation.

Table 1. Brands and models of devices used in the study

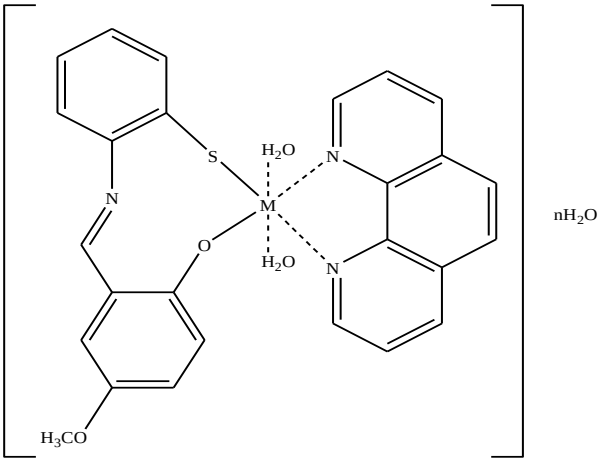
Device	Brands and models
FT-IR	Perkin-Elmer Spectrum 100 FTIR spectrophotometer (ATR)
Elemental Analysis	Thermo Scientific Flash EA 2000 CHNS
Molar Conductivity	ORION 4 STAR pH Conductivity Benchtop
Magnetic Susceptibility	Sherwood Scientific
UV-Vis	PG Instruments T80+UV/Vis spectrometer

2.2. Synthesis of the ligand (H_2L)

The ligand (2-((2-mercaptophenylimino)methyl)-4-methoxyphenol) (**H_2L**) was synthesized according to the literature (Muthu Tamizh et al., 2012).

2.3. General synthesis of complexes with H_2L and 1,10-phenanthroline

The complex was created by mixing the solution of $M(OAc)_2 \cdot nH_2O$ (0.25 mmol) (M=Cu, Ni and Co, n=1, 4, 4) in methanol and the ligand 2-((2-mercaptophenylimino)methyl)-4-methoxyphenol **(H_2L)** (0.065 g, 0.25 mmol) and 1,10-phenantrolin (**phen**) (0.045 g, 0.25 mmol) solution in chloroform/methanol (1:1) with 1:1:1 ligand(H_2L):metal:ligand(phen) mole ratio. The mixture used in the process was refluxed for 30 minutes. Filtered, washed with ice-cold methanol and water. The complex was purified from slowly vapor diffusion of Et_2O with a MeOH solution of complexes and vacuum-dried, the end product was produced. (Figure 1).



M: Cu(II), n: 1; Ni(II), n:2; Co(II), n:3

Figure 1. General structure of mixed ligand complexes

***[Cu(L)phen(H₂O)₂]· H₂O (L-Cu)*:** The synthesized mixed ligand copper complex (**L-Cu**) is brown in color and purified in methanol (Figure 1). Yield: 0.097 g (70 %). 197.3 °C decomp. IR, (ATR) *ν*, cm^{-1}: 3396 (O-H), 3061 $(C\text{-}H)_{arm.}$, 2935 $(C\text{-}H)_{alp.}$, 1620 (C=N), 553 (M-N), 457 (M-O); UV-Vis (DMF) λ_{max} (Abs): 305 (0.624), 335 (0.374), 420 (0.315), 460 (0.355), 490 (0.22), nm; μ_{eff}: 1.97 B.M. Λ_M (DMF, 10^{-3} M, µS/cm): 9.04. Anal.Calc. For $C_{26}H_{25}CuN_3O_5S$ (555.1); C, 56.26; H,4.54; N, 7.57; S, 5.78. Found: C, 55.80; H, 3.96; N, 7.08; S, 5.24 %.

***[Ni(L)phen(H₂O)₂]·2H₂O (L-Ni)*:** The synthesized mixed ligand copper complex (**L-Ni**) is reddish-brown in color and purified in methanol (Figure 1). Yield:0.115 g (80 %). m.p:154-155 °C. IR, (ATR) *ν*, cm^{-1}: 3385 (O-H), 3060 $(C\text{-}H)_{arm.}$, 2974 $(C\text{-}H)_{alp.}$, 1627 (C=N), 536 (M-N), 457 (M-O); UV-Vis (DMF) λ_{max} (Abs): 305 (0.619), 335 (0.301), 420 (0.218), 465 (0.300), 495 (0.223), 515 (0.164) nm; μ_{eff}: 2.92 B.M. Λ_M (DMF, 10^{-3} M, µS/cm): 2.46. Anal.Calc. for $C_{26}H_{27}N_3NiO_6S$ (568.27); C, 54.95; H, 4.79; N, 7.39; S, 5.64. Found: C, 55.20; H, 4.52; N, 6.54; S, 5.29 %.

***[Co(L)phen(H₂O)₂]·3H₂O (L-Co)*:** The synthesized mixed ligand copper complex (**L-Co**) is dark brown in color and purified in methanol (Figure 1). Yield: 0.121 g (82%). m.p: 217.7 °C decomp. IR, (ATR) *ν*, cm^{-1}: 3358 (O-H), 3057 $(C\text{-}H)_{arm.}$, 2976 $(C\text{-}H)_{alp.}$, 1620 (C=N), 549 (M-N), 459 (M-O); UV-Vis (DMF) λ_{max} (Abs): 330 (1.205), 305 (1.290), 315 (1.547) nm; μ_{eff}: 2.84 B.M. Λ_M (DMF, 10^{-3} M, µS/cm): 3.24. Anal.Calc. for $C_{26}H_{29}CoN_3O_7S$ (586.52); C, 53.24; H, 4.98; N, 7.16; S, 5.47. Found: C, 52.90; H, 4.27; N, 6.52; S, 4.70 %.

3. Results and discussion

3.1. Synthesis and characterization of compounds

The Schiff base (H_2L) ligand has synthesized by condensing 2-hydroxy-5-methoxybenzaldehyde with 2-aminobenzenethiol in equimolar ratio 1:1 in MeOH (Muthu Tamizh et al., 2012). Mononuclear metal(II) coordination complex was formed as a result of the reaction between Schiff base (H_2L) and 1, 10-phenathroline with $M(OAc)_2.nH_2O$ (M:Cu, Ni, Co, n:1,4,4). The mixed ligand metal complex elemental analysis results are consistent with calculated values, demonstrating that the complexes have a metal-ligand ratio of 1:1:1 (H_2L:Metal:phen). O, N, and S-tridentate mode coordination of the Schiff base ligand with the Cu(II) ion is achieved. In chloroform, DMSO, MeOH, DMF, acetonitrile, and THF, the ligands are soluble. At room temperature, the recently created mixed ligand complexes are extremely solid-state stable. Although the ligand and complexes were crystallized, the desired single crystal could not be obtained. Based on the results of the elemental analysis, we have calculated that the general formula for mixed ligands is $[ML(phen)H_2O]\cdot nH_2O$, where L is the anion of H_2L. As indicated by the molar conductance measuring for 10^{-3} M solutions in DMF falling in the range of 2.46-9.04 µS/cm, these complexes display non-electrolytic activity (Sönmez et al., 2014; Maravalli et al., 1999).

When the mixed ligand metal complexes' FT-IR spectra are studied, a broad band between 3396-3358 cm^{-1} is observed, which is thought to be the *ν*(OH) stretching band. The stretching band of azomethine (C=N) in the complexes was observed between 1620-1626 cm^{-1}.The azomethine band of ligand was revealed at 1633 cm^{-1} (Kesavan et al., 2012). Actually, in the mixed ligand complexes, this band was moved to lower (7-13 cm^{-1}) wavenumbers, proving that azomethine nitrogen is involved in the coordination of metal ions (Dede et al., 2018). When the complexes' FT-IR spectra were analyzed, it was found that the *ν*(M-N) vibrations were between 553-536 cm^{-1} and the *ν*(M-O) vibrations were between 459-457 cm^{-1} (Sönmez et al., 2006; Sönmez and Şekerci, 2003). (Figure 2-5)

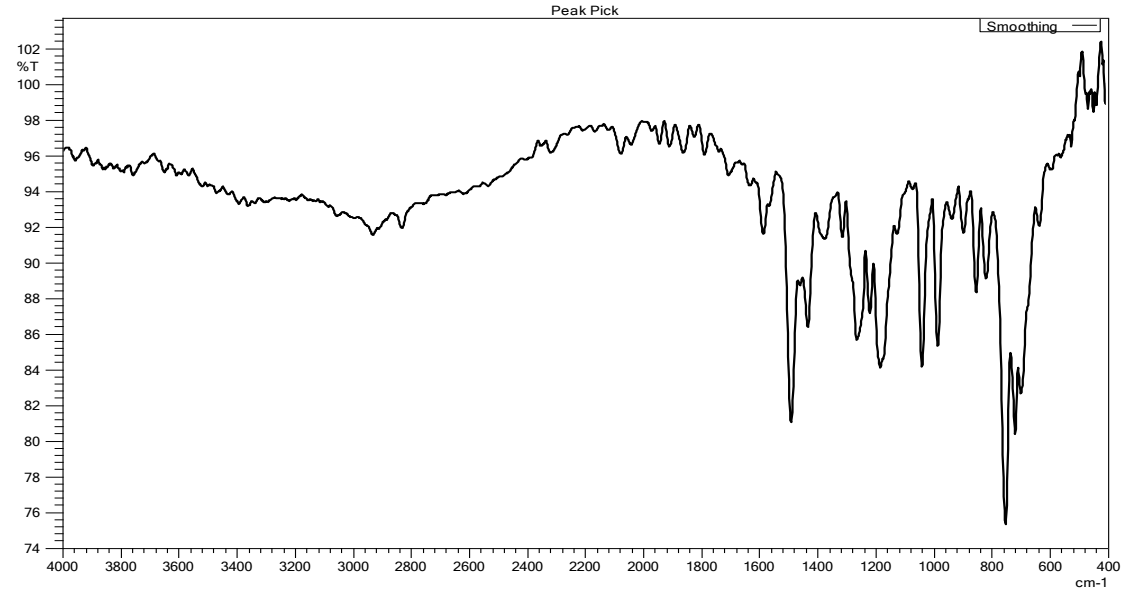


Figure 2. FT-IR spectrum of the ligand (H_2L)

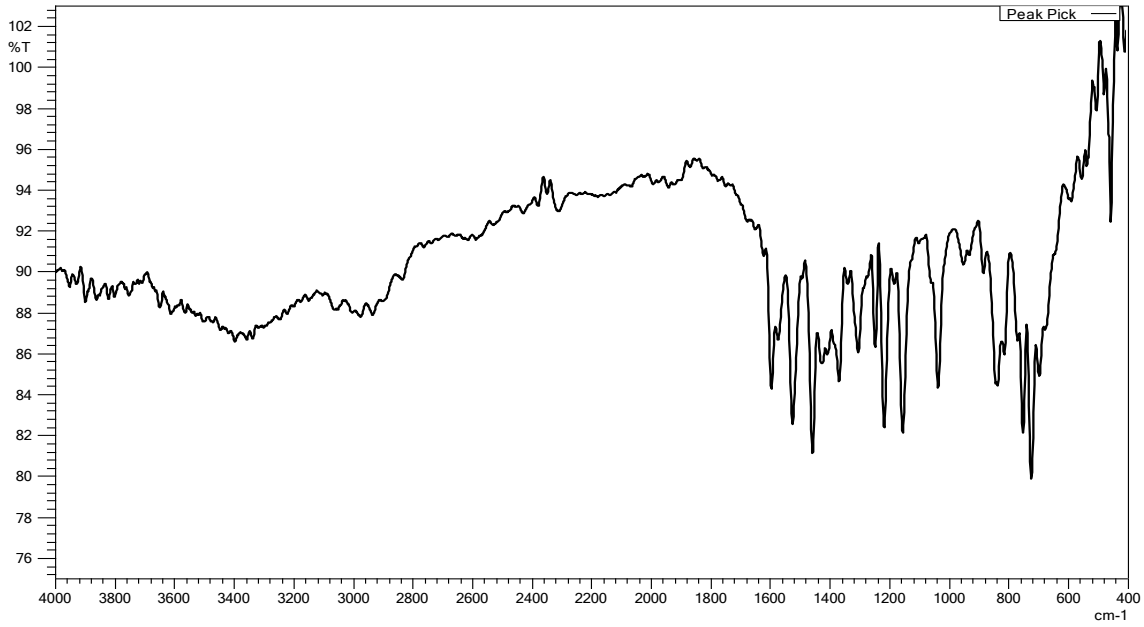


Figure 3. FT-IR spectrum of the L-Cu

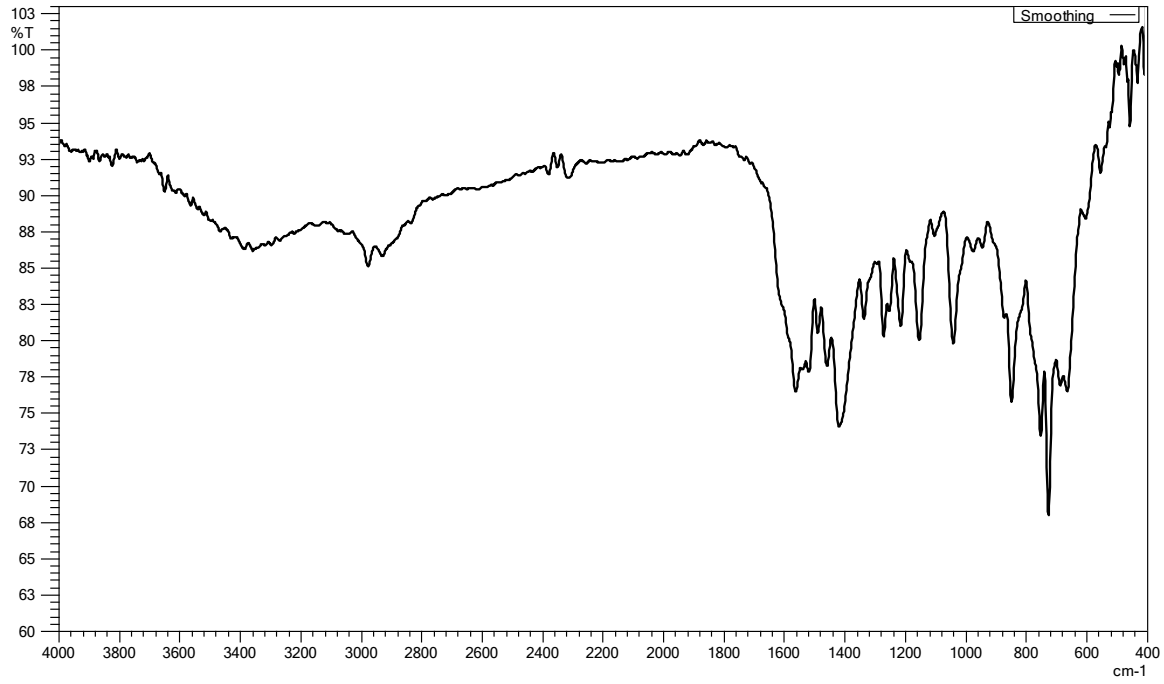


Figure 4. FT-IR spectrum of the L-Ni complex

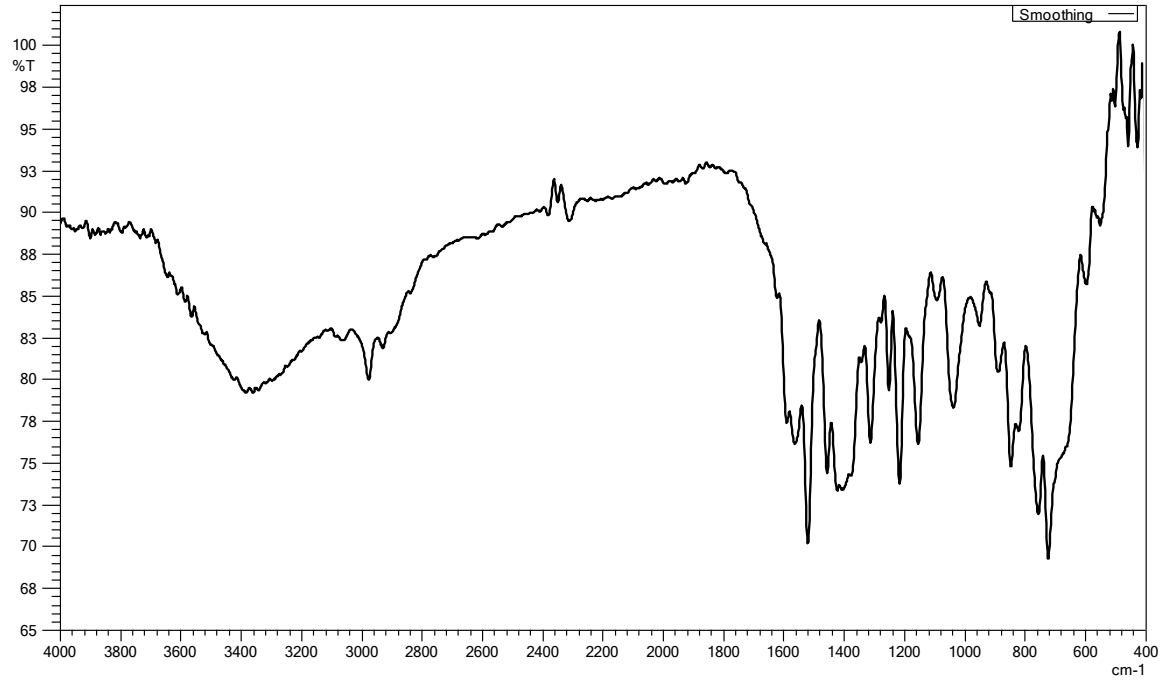


Figure 5. FT-IR spectrum of the L-Co complex

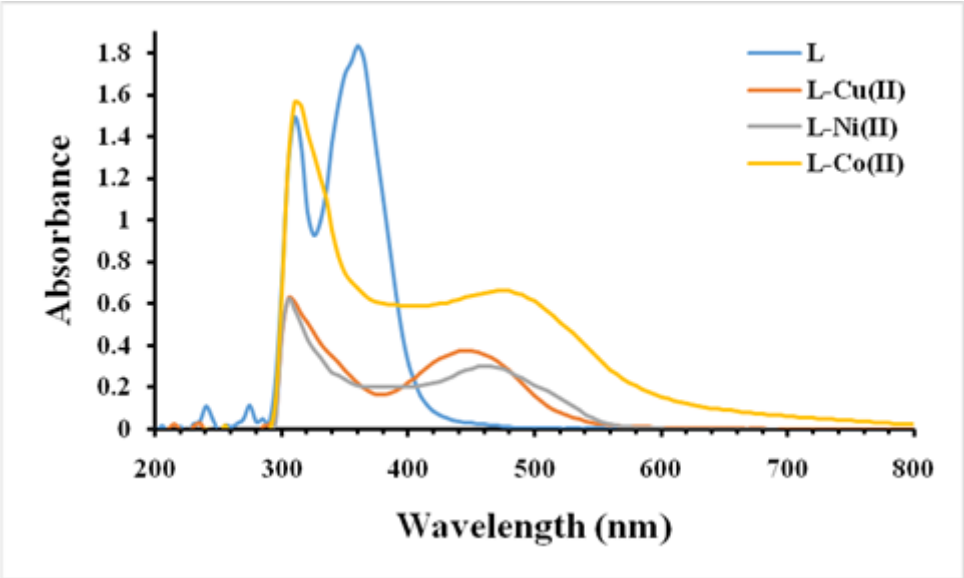


Figure 6. UV-Vis spectrum of compounds

In the N,N-dimethyl formamide solvent at a concentration of $2x10^{-5}$ M, UV-Vis spectra in the region of 190-1100 nm were obtained. Examining the produced mixed ligand complexes' findings and UV-Vis spectra, it is thought that the transitions in the range of 305-335 nm in the complexes are the π-π* transitions of the phenyl and 1,10-phenanthroline rings (Figure 6) (Sönmez and Şekerci, 2007). In addition, the absorption bands in the 335-365 nm range are thought to relate to the n-π* transitions of the 1,10-phenantroline ring and the azomethine group. New bands appearing in the complexes in the 420-515 nm range were observed as charge transfer transitions. This indicates complex formation (Goorchibeygi et al., 2022). At 298 K, the complexes' magnetic susceptibility was measured. Considering the magnetic susceptibility values, the L-Cu, L-Ni and L-Co complexes were recorded as 1.97, 2.92 and 2.84 BM, respectively. The magnetic susceptibility value for L-Cu was found to be 1.97 BM. Octahedral and mononuclear structures have been proposed for the L-Cu complex (Ekmekcioglu et al., 2015; Gülcan and Sönmez, 2011; Salib et al., 2003). The measured value of 2.92 BM for the nickel complex (L-Ni) is in the range of 2.8-3.2 BM given in the literature, suggesting that the structure may be octahedral. (Salib et al., 2003). When the magnetic susceptibility data of the L-Co complex is examined, It has been noted that this value is lower than that of the octahedral structures in the literature. While the μ_{eff} value of high spin cobalt complexes from d^7 elements is expected to be 3.87 BM according to the formula $(n(n+2))^{1/2}$ (n=number of unpaired electrons), the magnetic susceptibility value of the cobalt complex was found to be 2.84 BM. This shows that the Co(II) complex has antiferromagnetic properties (Min et al., 2007; Sogukomerogullari and Başaran, 2022). It is thought that the mixed ligand cobalt complex (L-Co) has an octahedral structure (Sogukomerogullari et al., 2019; Sogukomerogullari and Başaran, 2022).

4. Conclusions

The nature of mixed ligand complexes is highlighted in this work. In the work, novel mixed Co(II), Ni(II), and Cu(II) complexes were created using the ligand (1,10-phenantroline) and (2-((2-mercaptophenylimino)methyl)-4-methoxyphenol). Utilizing a variety of techniques, including elemental analysis, molar conductivity UV-Vis, magnetic susceptibility, and FT-IR the structures of the produced compounds were identified. The synthesized complexes are original and consistent with literature studies. The synthesized complexes were determined to have a H_2L:metal:phen (1:1:1) cytoichiometric ratio. The complexes are thought to be in six coordinated octahedral structures. Considering the application areas of mixed ligand Schiff base complexes in the literature, it is thought that this study will contribute to aiding research in the future.

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Author contribution

Hatice Gamze Sogukomerogullari: Supervision, Data curation, Formal analysis, Investigation, Methodology, Writing–original draft, Validation, Conceptualization. Ahmet Oral Sarıoğlu: Investigation, Methodology, Writing–original draft, Validation, Conceptualization

Declaration of ethical code

The author of this article declares that the materials and methods used in this study do not require ethical committee approval and/or legal-specific permission.

Conflicts of interest

The author declares that there is no conflict of interest.

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