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Abstract

Schiff bases are condensation product compounds obtained from the reaction of carbonyl group compounds with primary amines. The C=N bond formed due to the reaction is called the azomethine bond or imine bond. Coordination compounds formed by Schiff bases with metals have broad application areas and play essential roles as catalysts, biological agents, analytical reagents, materials, and environmental solutions. These compounds' various chemical and physical properties increase their importance in basic science research and industrial applications. In this study, Schiff base ligands (3,5) were synthesized from the reaction of vanillin, an aldehyde compound, with primary amines in an acidic medium. The structure of the synthesized Schiff base ligands was characterized by ^{1}H NMR, ^{13}C NMR, and FTIR spectroscopy, and new metal complexes (6,7) were synthesized from the reaction with cadmium metal salt ($Cd(NO_3)_2$). The structures of the synthesized metal complex compounds were characterized by elemental analysis, FTIR, and thermogravimetric analysis, and their properties were investigated. These findings suggest that it has the potential to be used in various technological and industrial applications.

Keywords: Schiff Base, Cadmium (II), Ligand, Metal Complex, Thermal Stability,

INTRODUCTION

German chemist Hugo Schiff synthesized Schiff bases in 1869[1]. Schiff bases are compounds obtained by condensing carbonyl groups of aldehydes or ketones with imine or azomethine groups under certain reaction conditions of primary amines (Figure 1)[2]. Schiff base reactions are highly efficient and easy reactions. Schiff bases are prone to form complexes by reacting with almost all metals thanks to the nitrogen atom in the imine group, phenoxyl hydrogen in the structure, sulfur atom in the thiol group, and electron-donating atoms of carboxyl groups [3-7].

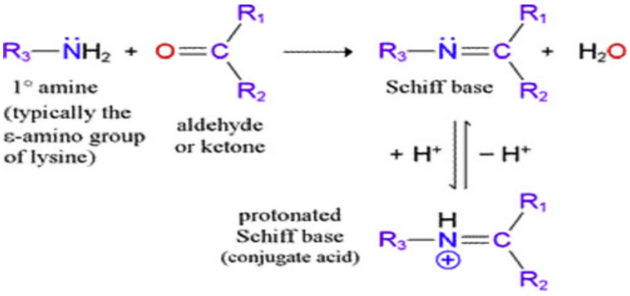


Figure 1. General scheme of Schiff base synthesis

Although there are many aldehyde and amine compounds, the stability of Schiff bases obtained from the reaction of these aldehyde compounds is different [8]. For the synthesized Schiff base ligands to be stable, the presence of a substituted group adjacent to the azomethine group and a second substitutable hydroxyl group increases the stability of the ligand[9]. For Schiff bases to show the best ligand properties, substituted groups such as -OH, $-NH_2$, -SH, and $-OCH_3$ should be attached to the imine group in the ortho state. Due to the structural and biological properties of Schiff bases, they were first studied in coordination chemistry with metal complexes by Pfeiffer in 1933 [10,11,12].

The structure of the complex formed by the ligand with metals is shaped by the metal salt, the mole ratio of the ligand and the metal salt, and the structure of the molecules. The stability of complexes formed by metals with multivalent ligands increases[13]. It is stated that the stability of Schiff bases is due to the Lewis base property due to the unshared electron pair on the nitrogen atom in the imine group, which forms stable compounds by coordinated covalent bonding with metal salts. Schiff bases are generally shown as RCH=N-Ar. R in the formula is aryl alkyl or alkyl substituents.

Schiff bases and the metal complexes they form have many uses. Metal complexes are pigment dyestuffs in textile dyeing since they show dyestuff properties. Depending on the structure of the groups in the benzene ring in the structure of Schiff bases, ligands showing inhibition properties are also used as inhibitors [14,15]. Schiff base ligands have roles in oxidizing biologically active molecules such as free oxygen, ascorbic acid, catechol, and amino acids by forming coordination compounds with metals[16,17]. Metal complexes of ligand compounds containing heterocyclic thio semicarbazides are used in health treatment due to their antitumor, bacterial, and antiviral properties[17]. In addition, it is known that platinum complexes show antitumoural activity and nitro and halo derivatives show both antimicrobial and antitumoural activity[18,19,20]. In studies with oxo-vanadium(IV) and oxo-vanadium(V) complexes of Schiff base ligands, it was observed that the compounds were particularly influential on plant pathogens Agrobacterium Tumefaciens and Helminthosporium Oryzae [21]. It is also known that ninhydrin and glycine derivative Schiff base metal (Co(II), Ni(II), Zn(II)) complexes are effective on Escherichia coli, Proteus Mirabilis, Staphylococcus Aureus and Streptococcus faecalis [22]. Ferrocene-based metal (II) Schiff base complexes were synthesized from ferrocenyl chalcone in a solvent-free medium and found antibacterial properties [23,24].

The complexes formed by Schiff bases with cadmium metal are important due to the special properties and application potential they provide in various fields. Here are some highlights of these complexes:

Catalytic Activity: Schiff base-cadmium complexes can be used as catalysts in organic synthesis. These complexes show high activity, especially in reactions such as oxidation, hydrogenation, and C-C bond formation [26].

Photophysical and Photochemical Properties: These complexes can be used in optoelectronic devices, photovoltaic cells, and light-emitting diodes (LEDs). Cadmium complexes can exhibit unique properties in light absorption and emission [27].

Therapeutic Applications: Some Schiff base-cadmium complexes are being investigated in the biomedical field as anticancer, antibacterial, and antifungal agents. The biological activity of these complexes allows their use in treating certain diseases [28].

Supermolecular Chemistry: Schiff bases and cadmium can create supermolecular structures and materials. Such structures are essential in molecular recognition, sensors, and materials science [29].

Real-World Environmental Applications: The use of cadmium-containing Schiff base complexes for the removal of heavy metal ions and environmental pollution is not just theoretical. These complexes hold the potential to revolutionize water treatment processes, making a tangible impact on our environment [30].

Coordination Chemistry and Structural Diversity: The complex structures of Schiff base and cadmium show diversity in coordination chemistry and form the basis for synthesizing new complexes. The structural properties of these complexes open the door to new research in chemistry and materials science [31].

For these reasons, the complexes formed by Schiff bases with cadmium metal play an essential role in academic research and industrial applications.

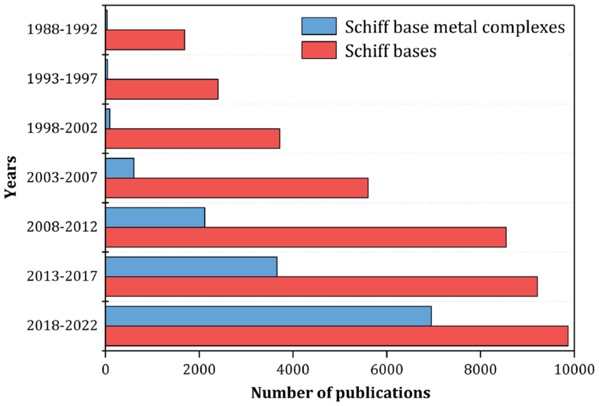


Figure 2. The data of studies with Schiff Bases between 1988 and 2022 [32]

MATERIALS AND METHODS

Materials: The chemicals o-vanillin, 3-amino-4-hydroxy benzene sulphonic acid, and 2-amino-4,5-dimethoxy benzoic acid were used as starting material in the synthesis of Schiff base ligand and metal salt ($Cd(NO_3)_2$) used in the synthesis of the metal complex. Sigma-Aldrich, ethyl alcohol, acetic acid, and methanol solvents used were supplied by Merck. The UV lamp, CAMAG Muttenz-Schweiz 29200, and melting point apparatus, Büchi SMP 20, were used to elucidate the melting points of the synthesized ligands [7]. Infrared spectra were taken from 400 to 4000 cm-1 using a Thermo Scientific Nicolet 6700 FTIR spectrometer. ^{1}H and ^{13}C NMR spectra of the synthesized Schiff bases (**3**,**5**) were obtained with a liquid Bruker 400MHz AV model NMR spectrometer with a 400 MHz operating frequency. The obtained metal complexes (**6**,**7**) were thermally analyzed using the TA Instruments brand and the Q600 SDT (Simultaneous DSC/DTA/TGA) model device. The percentage of elements in the complexes' structures was determined by the Leco brand Truspec Micro Elemental Device.

Metods: In a 100 mL reaction flask, the aldehyde compound was dissolved in 30 mL ethyl alcohol. Then, the amine compound was added at a molar ratio 1:1. The mixture was stirred until completely dissolved. While the reaction mixture was stirred under a cooler, 1-2 drops of acetic acid were added to the reaction flask to maintain the pH around 4-5. The reaction mixture was stirred under a cooler for 12 hours, and after precipitation with water, the solid fraction was obtained by filtration. The solid portion was dried, and FTIR spectroscopy was used to determine whether the reaction occurred [2,7,8]. Schiff base was added to a 100 mL reaction flask and 30 mL of methanol solvent was added to dissolve the Schiff base. $Cd(NO_3)_2$ metal nitrate salt was dissolved in ethanol solvent in a beaker and added to the reaction flask. The reaction mixture was stirred under reflux for 12 hours, and after precipitation with water, the solid was filtered and purified by washing in methanol solvent to obtain cadmium complexes [7,8,10].

SYNTHESIS AND ANALYSIS OF SCHIFF BASE

4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino) benzenesulfonic acid (3)

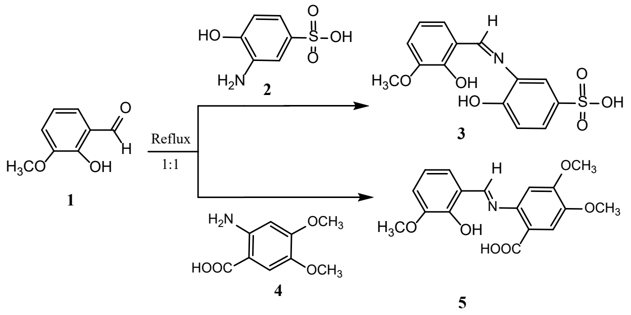
4- hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino) benzenesulfonic acid (**3**) ligand was synthesized from the reaction of o-vanillin (**1**) (0.5g, 3.3 mmol) with 3-amino-4-hydroxybenzenesulfonic acid (**2**) (0.6g, 3.3 mmol) (Scheme 1).

3: Orange solid. E.N: 284.1°C, Yield: 0.6 g (% 56.6); IR (KBr): v = 3665, 3250 (O-H), v = 3159, 3100, 3068 (C-H_{arom}), v = 2933, 2846 (C-H), 1644 (HC=N), 1610 (C=C), 1233 (C-O); ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.68,7.57, 7.48, 7.40, 7.32, 7.26, 7.14, 6.92 (m, 6H, CH_{arom}), 9.09 (s, 1H, N=CH_{imin}), 9.73 (s, 1H, S-OH), 10.28, 10.86 (s, 2H, C_{fenil}-OH), 3.86 (s, 3H, -OCH_3); ^{13}C-NMR (100 MHz, CDCl3): δ = 162.15 (C=NH), 152.09, 151.17, 148.82, 140.66, 127.33, 124.93, 122.96, 119.67 (C_{arom}), 56.55 (OCH_3).

(E)-2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxybenzoic acid (5)

(E)-2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxybenzoic acid (5) ligand was synthesized from the reaction of o-vanillin (1) (0.5g, 3.3 mmol) with 2-amino-4,5-dimethoxybenzoic acid (4) (0.65g, 3.3 mmol) (Scheme 1).

5: Yellow solid. E.N: 218.2°C.Yield: 0.95g (% 88). IR (KBr): v = 3542, 3136 (O-H), v = 3074, 3008 (C-H_{arom}), v = 2985, 2943, 2901 (C-H), 1687 (C=O), 1604 (HC=N), 1513 (C=C), 1215 (C-O); ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 10.28 (s, 1H, COOH), 8.92 (s, 1H, -NCH_{imin}), 7.46, 7.20, 7.07, 6.87 (m, 4H, CH_{phenyl}), 3.91, 3.87, 3.80 (m, 9H, -OCH_3); ^{13}C-NMR (100 MHz, CDCl3): δ = 167.49 (COOH), 162.06 (HC=N), 153.06, 150.98, 148.53, 142.38, 124.01, 120.59, 119.99, 115.76, 99.52 (C_{arom}), 56.55 (OCH_3).



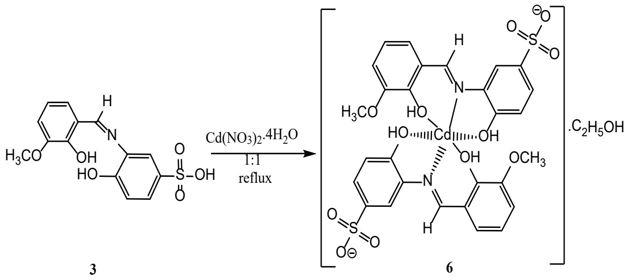
Scheme 1. Synthesis scheme of shiff base ligands

SYNTHESIS AND ANALYSIS OF CD(II) METAL COMPLEXES

bis(4-hydroxy-3-((2-hydroxy-3- methoxybenzylidene)amino) benzenesulfonato
-κO,κN,κO')cadmium(II) monoethanol (6)

Bis(4-hidroksi-3-((2-hidroksi-3-metoksibenziliden)amino) benzensulfonato-κO,κN,κO')cadmium (II) mono ethanol (6) complex was synthesized from the reaction of 4-hydroxy-3-((2-hydroxy-3-methoxy benzylidene)amino)benzenesulfonic acid (3) (1g, 3.1 mmol) ligand and Cd(NO3)2 (2) (0.95 g, 3.1 mmol) salt (Scheme 2).

6 : Orange solid. E.N: 372°C. Yield: 0.52g (% 20). IR (KBr): ν = 3542, 3136 (O-H), ν = 3074, 3008 (C-H_{arom}), ν = 2985, 2943, 2901 (C-H), 1636 (HC=N), 1494 (C=C), 1220 (C-O).

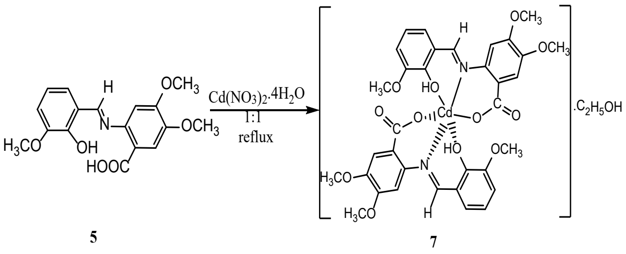


Scheme 2. Synthesis of bis(4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino)benzenesulfonato-κO,κN,κO')cadmium (II) monoethanol (6) complex

bis(2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxybenzoato-κO,κN,κO')cadmium(II) monoethanol (7)

bis(2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxybenzoato-κO,κN,κO') cadmium (II) monoethanol (7) complex was synthesized from the reaction of 4(E)-2-((2-hidroksi-3-metoksibenziliden)amino)-4,5-dimetoksibenzoik acit (5) (1 g, 3.1 mmol) ligand and $Cd(NO_3)_2$ (2) (0.96g, 3.1 mmol) salt (Scheme 3).

7 : Red solid. E.N: 219-202°C Yield: 0.56g (% 22). IR (KBr): ν = 3669, 3313 (O-H), ν = 3059, 3007 (C-H_{arom}), ν = 2964, 2937, 2835 (C-H), 1628 (C=O), 1543 (HC=N), 1362 (C=C), 1268 (C-O).



Scheme 3. Synthesis of bis(2-((2-hydroxy-3-methoxybenzylidene) amino)-4,5-dimethoxybenzoato-κO,κN,κO') cadmium (II) monoethanol (7) complex

Elemental Analysis of Cd(II) Metal Cation-Centred Complexes

According to the results of elemental analysis of metal-centered complexes formed by 4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino)benzenesulfonic acid (3) and (E)-2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxybenzoic acid (5) ligands with Cd(II) metal cation, the theoretical and experimental results of C, H, N, S data support each other. Although elemental analysis alone is insufficient, it was determined that complexes were obtained when supported by other analyses. Table 1 shows the results of the analyses.

Table 1. Elemental analysis data of Cd(II) metal cation-centred complexes

Complex Molecule	C (%)		H (%)		N (%)		S (%)	
	Exp.	Calc.	Exp.	Calc.	Exp.	Calc.	Exp.	Calc.
(**6**)$C_{30}H_{30}CdN_2O_{13}S_2$	45.12	44.87	4.48	3.77	3.40	3.49	8.08	7.98
(**7**) $C_{36}H_{38}CdN_2O_{13}$	52.79	52.01	5.43	4.68	3.50	3.42	-	-

Thermal Analysis of Cd(II) Metal Cation Centred Complexes

Thermal analysis curves (TGA/DTA/DrTG) of the coordination compounds of Shiff base ligands with Cd(II) metal cations as center atoms are given in Figure 3. All data on the thermal decomposition steps and decomposition products generated from the thermal analysis curves are summarised in Table 2. The complex (I) with 4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino)benzenesulfonic acid (3) ligand has five degradation steps, while the complex (II) containing (E)-2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxy benzoic acid (5) ligand was found to degrade in four steps.

It is suggested that ethyl alcohol, used as the synthesis solution in the synthesis of the complexes, binds to the structures by hydrogen bonds and settles outside the coordination sphere. The bonding of ethyl alcohol to the outside of the coordination sphere by hydrogen bonding occurs due to the formation of hydrogen bonds, steric hindrances, solvent interactions, and electron density distribution. These interactions affect the behavior and stability of Shiff base complexes in solution.

Ethyl alcohol can form hydrogen bonds through the hydrogen atom in the -OH group. This hydrogen bond interacts with the appropriate electron pair donors near the Shiff base complex. These bonds usually form outside the coordination sphere because ethyl alcohol is a solvent that does not bind directly to the coordination center [33-34]. Our suggestion is supported by the fact that the weight losses observed in the first decomposition steps of both complexes (43-120°C and 37-125°C, respectively) are consistent with the theoretical and experimental weight losses of ethyl alcohol (For structure 6, exp.: 5.02%; theo. 5.73% and For structure 7, exp.: 5.10%; theo. 5.62%). The subsequent degradation steps for both structures include data on the thermal degradation of the organic ligands (Table 2).

The conclusion that CdO remains in the reaction vessel as the final residual product of the thermal decomposition of complex six at 881°C is also in agreement with the theoretical and experimental weight losses (exp.: 16.96%; theo.: 15.99%). Similarly, the conclusion that CdO oxide remained as the final decomposition product as a result of the thermal decomposition of complex seven at 912°C was inferred from the agreement between the theoretical and experimental weight losses of the final decomposition product (exp.: 16.58%; theo.: 15.68%). The data that the final decomposition products were CdO were also supported by powder XRD patterns. A difference of approximately 1% was observed between the theoretical and experimental weight losses of both final decomposition products. This was attributed to the lack of sufficient oxygen in the structures during thermal degradation in an inert nitrogen atmosphere and to the fact that the carbon residue of the organic ligand could not complete combustion, and some of it was deposited on the metal oxide residues in the form of carbonized carbon. The black color of the final decomposition products, which were expected to be white, also supports this interpretation.

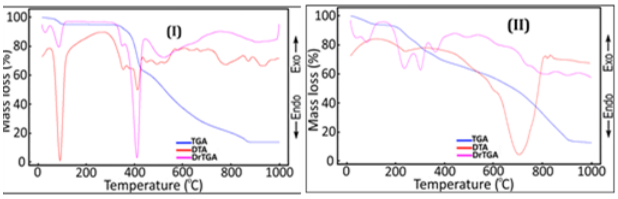


Figure 3. Thermal curves of Cd(II) metal cation-centered Schiff base complexes.

RESULT AND DISCUSSION

The characteristic structures of the synthesized Schiff base ligands were elucidated by Infrared Spectroscopy and ^{1}H and ^{13}C-NMR spectra. Ligands with an imine group were obtained from the reaction of aldehyde compounds with a carbonyl group in Schiff bases and primary amines in the essential medium. The absorption stretching band of the imine group of 4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino) benzenesulfonic acid (3) ligand was detected at 1644 cm-1 in infrared spectroscopy, while the stretching band of (E)-2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxybenzoic acid (5) ligand was detected at 1604 cm^{-1}. In addition, in the ^{1}H-NMR spectrum, the chemical shift of the imine group proton of 4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino) benzenesulfonic acid (3) was detected at 9.09 ppm and the chemical shift of E)-2-((2-hydroxy-3-methoxybenzylidene) amino)-4,5-dimethoxy benzoic acid (5) at 8.92 ppm. The occurrence of imine groups in different regions between the two ligands is due to the different electron affinity of the sulfonyl and carbonyl groups in the structure of the ligands. The carbonyl group is generally more electronegative than the sulfonyl group because the difference in electronegativity between carbon and oxygen is slightly higher than that between sulfur and oxygen. Therefore, the imine group of the ligand with the sulfonyl group shows a chemical shift in the lower field. The ligand 4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino)benzenesulfonic acid (3) is orange in color and has a higher melting point than E)-2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxy benzoic acid (5) containing a carbonyl group. Since compounds with a sulfonyl group have higher polarity, the sulfonyl group gives the compounds a high dipole moment. Increasing the dipole-dipole interactions between the molecules can raise the melting point. In addition, hydrogen bonds also increase the melting point thanks to groups that can form hydrogen bonds in the compound. The melting point of 4-hydroxy-3-((2-hydroxy-3-methoxybenzylidene)amino)benzenesulfonic acid (3) is 284°C, while the melting point of E)-2-((2-hydroxy-3-methoxybenzylidene)amino)-4,5-dimethoxybenzoic acid (5) is 218°C. When the physical properties of Cd(II) metal cation complexes are examined, metal complex six is orange colored, and metal complex seven is red colored. As in the ligands, the melting point of metal complex six is 372°C due to the presence of a sulfonyl group in its structure. In contrast, the melting temperature of complex seven is lower at 219°C due

Tablo 2. Thermal decomposition data of Cd(II) metal cation-centered Schiff base complexes.

Compounds		Temp. range (˚C)	DTA_{max} (˚C)	Removing group	Mass Loss (%)				Remaining Product (%)	Colour
					Exp.	Theo.	Exp.	Theo.		
(**6**) $[Cd(C_{14}H_{12}NO_6S)_2](C_2H_5OH)$										white
$C_{30}H_{30}CdN_2O_{13}S_2$	1	43-12	106	C_2H_5OH	5.02	5.73				
803,1 g/mol	2	310-384	365	$2CH_3$	4.08	3.74				
	3	385-441	393;424	$2C_7H_4O$	26.07	25.93				
	4	442-550	501;532	$2SO_2$	16.28	15.96				
	5	552-885	644;680;785;875	$C_6H_5O;C_6H_5;2NO_2$	31.59	32.65	16.96	15.99	CdO	black
(**7**) $[Cd(C_{17}H_{16}NO_6)_2](C_2H_5OH)$										pale-white
$C_{36}H_{38}CdN_2O_{13}$	1	37-125	46;91	C_2H_5OH	5.10	5.62				
819,11 g/mol	2	169-317	238;302	$6CH_3O$	23.22	22.72				
	3	319-575	364;488	$2C_7H_2O$	23.96	24.92				
	4	576-780	618;699;733	$C_7H_5;NO_2;NO$	31.14	31.04	16.58	15.68	CdO	black

to the presence of a carbonyl group in its structure and the absence of a sulfonyl group. Considering the thermal stability, the thermal stability of compound six is higher as complex six starts to decompose at 43°C while complex seven starts to decompose at 37°C. Experimental and theoretical data of C, H, and O ratios in elemental analyses support the structural characterization of the metal complexes.

The magnetic susceptibility of cadmium metal is shallow and negative, indicating that it is a diamagnetic material. Since the diamagnetic property of cadmium means that magnetic fields weakly repel it, the magnetic susceptibilities of the two metal complexes obtained were not examined. In the literature, magnetic susceptibility analysis of coordination compounds made with Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, and Zn^{2+} metal cations is carried out due to their high magnetic susceptibility, but not in coordination compounds formed with Cd^{+2} cation.

The nitrogen atom of the imine group in metal complexes has four bonds. Four bonding of the nitrogen atom in the imine group in metal complexes of Schiff bases can reduce nitrogen's stability by reducing its electron density, but this may vary depending on the complex's overall geometric and electronic properties. Metal-ligand interactions and ligand field effects are important factors determining the effect of nitrogen quadruple bonding on stability. The nitrogen atom usually forms three bonds, each carrying one free electron pair. The formation of a fourth bond requires nitrogen to use this free electron pair, which can affect nitrogen's electron density and, hence, stability. The formation of a fourth bond can cause the formal charge of nitrogen to become positive, reducing the electron density and lowering stability [35,36].

Schiff bases containing a sulfonyl group are generally more stable and chemically resistant. However, Schiff bases containing carboxy groups are more differentiated by specific reactions and complex formation capabilities. Schiff bases containing carboxy groups are primarily used in analytical chemistry and complex formation processes. In contrast, those containing sulfonyl groups are generally more stable, which is a difference in finding a wide range of applications.

CONCLUSION

Schiff base syntheses and coordination compounds formed with metals have different properties and application areas, which increases the importance of Schiff bases. Although there are many coordination compounds made with Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, and Zn^{2+} metal cations in the literature, it is an essential study in terms of introducing new coordination compounds to the literature with the new Schiff bases obtained and forming a complex with Cd^{+2}.

When Schiff base ligands are combined with cadmium(II) salt, such as cadmium nitrate, the nitrogen of the azomethine group forms a coordinative bond with the cadmium ion, binding to organic compounds. The stability and high coordination of these complexes are further enhanced if the Schiff base contains additional donor atoms, such as -OH or another nitrogen atom, which also bind to the cadmium ion. This robust bonding pattern instills confidence in the potential of these Schiff bases in forming stable and highly coordinated complexes.

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