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s-Triazin İçeren Çok Dişli Schiff Bazlarının Sentezi ve Karakterizasyonu: Bazı Geçiş Metali Komplekslerinin Araştırılması

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ÖZET

Bu çalışmada ilk olarak sırasıyla siyanürik klorür ve hidrokinon veya 1,4-fenilendiaminin reaksiyona sokulmasıyla iki bazik ara ürün elde edildi. Bu bileşiklerin 4-hidroksibenzaldehit ile reaksiyonundan EBA1 ve EBA2 elde edildi. Bu bileşiklerin sırasıyla 4-aminobenzoik asit ve 5-aminoizoftalik asit ile reaksiyona sokulmasıyla EBL1-EBL4 olarak kodlanan dört hedef ligand elde edildi. Elde edilen bu ligandlar, FTIR, elementel analiz, ^{1}H ve ^{13}C NMR yöntemleriyle karakterize edildi. Elde edilen hedef ligandların $[M(Salen/Saloph)]_2O$ (M= Cr^{3+} ve Fe^{3+}) ile reaksiyonu sonucu [M(Salen/Saloph)] başlıklı kompleksler elde edildi. Bu kompleksler elementel analiz ve FTIR kullanılarak karakterize edildi. Komplekslerin manyetik özellikleri araştırıldı. Komplekslerin termal dayanıklılığı TGA tekniği kullanılarak belirlendi. Çalışmanın sonunda termal olarak kararlı ve paramanyetik özelliklere sahip 6 adet tetrapodal ligand ve 16 adet tetrapodal kompleks sentezlenmiş ve karakterize edilmiştir.

Synthesis and Characterization of Multidentate Schiff Bases Containing s-Triazine: Investigation of Their Some Transition Metal Complexes

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ABSTRACT

In this paper, two basic mid-products were obtained by reacting cyanuric chloride and hydroquinone or 1,4-phenylendiamine, respectively. EBA1 and EBA2 were obtained from the reaction of these compounds and 4-hydroxybenzaldehyde. Four target ligands encoded as EBL1-EBL4 were obtained by reacting these compounds with 4-aminobenzoic acid and 5-aminoisophthalic acid, respectively. These obtained ligands were characterized by FTIR, elemental analysis, ^{1}H and ^{13}C NMR and methods. The obtained target ligands were reacted with $[M(Salen/Saloph)]_2O$ (M= Cr^{3+} and Fe^{3+}) to obtain [M(Salen/Saloph)] capped complexes. These complexes were characterized using elemental analysis, and FTIR. The magnetic properties of the complexes were investigated. The thermal endurance of the complexes was determined using the TGA technique. At the end of the study, 6 tetrapodal ligands and 16 tetrapodal complexes, which are thermally stable with paramagnetic properties, were synthesized and characterized.

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INTRODUCTION

Schiff bases are defined as compounds formed as a result of the condensation reaction between primary amines and aldehydes or ketones, containing a carbon-nitrogen double bond (C=N) in their structure. This synthesis was first performed by German chemist Hugo Schiff in 1869. In the 1930s, Pfeiffer later utilized them as ligands. Schiff bases are also known as azomethines or imines [1]. The formation of Schiff bases from the reaction between primary amines and carbonyl compounds (aldehydes or ketones) occurs in two main steps. In the first step, a condensation reaction occurs between the primary amine and the carbonyl group, resulting in the formation of a carbinolamine intermediate. In the second step, this intermediate undergoes dehydration to produce the Schiff base [2]. Schiff bases exhibit different properties depending on the substituents attached to the imine group in their structure. Electronegative substituents on the nitrogen atom enhance the stability of azomethine. Schiff bases are more stable against alkali-containing groups, but they undergo hydrolysis in a low pH range. Upon hydrolysis, they convert to carbonyl and amine compounds. If there is an electronegative amine with an unshared electron on the nitrogen, the reaction is completed entirely. In this case, they are isolated with high efficiency since hydrolysis does not occur. The inductive effect is another factor affecting the stability of azomethines [3]. Schiff bases are preferred due to their low cost, ease of synthesis, and their highly stable chemical and thermal properties. They are used in the cosmetic industry, plastic industry, pesticides, chemotherapy in medicine, dye industry, electronics, analytical chemistry, polymer synthesis, as photographic chemicals, and for corrosion prevention in metals [4-6]. Medicines and many natural compounds consist of heterocyclic ring structures. In the field of chemistry, especially in inorganic and organic chemistry, there is a greater need for heterocyclic compounds, and they play an effective role in most synthesized compounds. Heterocyclic compounds are obtained as a result of the substitution of carbon atoms in the structure with heteroatoms such as sulfur, nitrogen, and oxygen. s-Triazines are also compounds with heterocyclic structures. s-Triazine compounds are formed by the substitution of three carbon atoms in the benzene ring with nitrogen atoms [7, 8]. 1,3,5-Triazine stands out as a captivating chemical scaffold in medicinal chemistry endeavors, owing to its diverse array of biological roles, including antimicrobial, antifungal, antimalarial, carbonic anhydrase inhibition, and anticancer properties [9-11]. Schiff bases with an s-triazine core demonstrate a range of biological activities, including anticancer, antimycobacterial, antibacterial, antidepressant, analgesic effects, and enzyme inhibition. Additionally, derivatives of 1,3,5-triazine have demonstrated the ability to bind to and regulate estrogen receptor alpha (ERα) and estrogen receptor beta (ERβ) [12,13].

In this study, we aimed to increase the thermal and electrical properties of such compounds to a higher level by synthesizing s-triazine-based supramolecular Schiff base ligands. For this reason, salen/saloph compounds have been used because they can coordinate well with other ligands. Starting from 2,4,6-trichloro-1,3,5-triazine, tetrapodal s-triazine-based Schiff base compounds containing carboxyl groups were synthesized. Salen/saloph initial complexes and capping complexes were obtained from these synthesized compounds, and the magnetic and thermal behaviors of the resulting complexes were examined.

EXPERIMENTAL

Apparatus and reagents

The ^{1}H and ^{13}C NMR analyses of the ligands were performed using a Bruker AvanceCore NMR spectrometer operating at 400 MHz. The ^{1}H and ^{13}C NMR spectra of the ligands were recorded in DMSO-d_6. Elemental analyses (C, H, N) of the compounds were carried out using a LECO 932 CHNS analyzer. The FTIR spectra of the synthesized complexes were recorded using a Thermo Nicolet iS5 FTIR spectrometer equipped with an ID7 ATR accessory. The metal contents of each complex were determined on a Varian, Vista AX CCD Simultaneous Model Inductively Coupled Plasma Atomic

Emission Spectrophotometer (ICP-AES). Magnetic susceptibility measurements of the complexes were performed using the Gouy method on a Sherwood Scientific MX Gouy device, with Hg[Co(SCN)4] as the calibrant. The effective magnetic moments, μ_{eff}, per metal atom were calculated from the expression: $\mu_{eff} = 2.84\sqrt{X_M}T$ B.M., where X_M is the molar susceptibility. TGA analyses of the compounds were conducted using a HITACHI STA7300 Thermal Analysis System.

Cyanuric chloride (Merck), 4-hydroxybenzaldehyde (Sigma), p-phenylenediamine (Sigma), 5-amino isophthalic acid (Sigma), 4-aminobenzoic acid (Sigma), hydroquinone (Sigma), potassium carbonate (Sigma), sodium carbonate (Sigma), sodium sulfate (Sigma), and solvents including acetone (Merck), benzene (Merck), ethyl acetate (Merck), ethanol (Sigma), methanol (Sigma), 1,4-Dioxane (Merck), N,N-dimethylformamide (Merck) were purchased from the mentioned companies. $[(MSalen/Saloph)_2O]$ (M = Cr^{3+} and Fe^{3+}) initial complexes, which are used in complex formation reactions and called ligand complexes, were synthesized and used according to the literature [14].

Synthesis of 4,4',4'',4'''-(((1,4-phenylenebis(oxy))bis(1,3,5-triazine-6,2,4-triyl))tetrakis(oxy))tetrabenzaldehyde (EBA1) and 4,4',4'',4'''-(((1,4-phenylenebis(azanediyl)) bis(1,3,5-triazine-6,2,4-triyl))tetrakis(oxy))tetrabenzaldehyde (EBA2)

EBS1 (2.03 g, 5.00 mmol) or EBS2 (2.02 g, 5.00 mmol) and 4-hydroxybenzaldehyde (2.69 g, 22.00 mmol) were weighed and placed into a 100 mL flask. Approximately 5.00 g of Na_2CO_3 and 50 mL of benzene were added. The mixture was stirred at 70°C under reflux for 24 hours. Then, the solid sodium carbonate residue was filtered under vacuum through filter paper. 20 mL of hot ethyl acetate was added twice, and the solid residue was discarded. The remaining liquid portion was washed twice with a 10% sodium carbonate solution and twice with distilled water. Sodium sulfate was added and the mixture was left overnight. The sodium sulfate was filtered out, and the organic solvent was evaporated. The resulting solid was recrystallized from ethanol. Compound EBA1 was elucidated using FTIR, ^1H, and ^{13}C NMR spectroscopy and elemental analysis method.

EBA1: Yellow solid; Yield: 78%; M.P.: 265-268°C; FTIR (ATR, cm^{-1}): 1698 (C=O), 1585 ($C=N_{trz}$), 1500, 1424 ($C-C_{arom}$), 1296 ($C-O_{phen}$); ^1H NMR δ (ppm): 9.87 (s, 4H), 7.84 (d, 8H), 7.15 (d, 8H), 7.07 (d, 4H); ^{13}C NMR δ (ppm): 192.10 (C9), 184.90 (C4), 177.50 (C3), 161.10 (C5), 150.01 (C2), 132.50 (C7), 123.30 (C1), 121.80 (C6), 116.10 (C8); Calculated/Found Analysis: C: 64.17/64.13, H: 3.23/3.21, N: 11.23/11.22, O: 21.37/21.33

EBA2: Yellow solid; Yield: 73%; M.P.: 184-187°C; FTIR (ATR, cm^{-1}): 3205 ($N-H_{str}$), 3069 ($C-H_{arom}$), 1694 (C=O), 1591 ($N-H_{bend}$), 1562 ($C=N_{trz}$), 1510, 1499, 1456, 1418 ($C-C_{arom}$), 1295 ($C-O_{phen}$), 1205 ($C-N_{str}$); ^1H NMR δ (ppm): 9.87 (s, 4H), 7.85 (d, 8H), 7.15 (d, 8H), 6.40 (d, 4H), 4.74 (s, 2H); ^{13}C NMR δ (ppm): 192.10 (C9), 184.90 (C4), 171.90 (C3), 161.20 (C5), 133.20 (C8), 131.50 (C7), 128.80 (C2), 122.00 (C6), 118.70 (C1); Calculated/Found Analysis: C: 64.34/64.33, H: 3.51/3.49, N: 15.01/15.00, O: 17.14/17.12.

General synthesis of target ligands (EBL1-EBL4)

A mixture of EBA1 or EBA2 (1.25 mmol) with 4-aminobenzoic acid or 5-aminoisophthalic acid (5.00 mmol) was dissolved in 50 mL of ethanol and 25 mL of DMF (N,N-dimethylformamide). The solution was then heated under reflux at a temperature of 85-90°C for 24 hours, while the reaction progress was monitored using thin-layer chromatography. Upon completion, the excess solvent was removed by evaporation, and the resulting solid product was collected by filtration. This crude product was further purified by recrystallization from an ethanol-DMF mixture. The final compound, labeled as EBL1, was characterized using elemental analysis, FTIR, and both ^1H and ^{13}C NMR spectroscopy.

EBL1: Yellow solid; Yield: 72%; M.P.: 334-336°C; FTIR (ATR, cm^{-1}): 1682 (C=O), 1592 ($C=N_{im}$), 1564 ($C=N_{trz}$), 1359 (COO^-), 1285 ($C-O_{phen}$); ^1H NMR δ (ppm): 10.60 (s, 4H), 8.69 (s, 4H), 8.24 (d, 8H), 7.79 (d, 8H), 7.60 (d, 8H), 7.08 (d, 8H), 7.01 (d, 4H); ^{13}C NMR δ (ppm): 184.90 (C4), 176.20 (C3), 168.90 (C14), 160.01 (C9), 157.50 (C10), 155.90 (C5), 149.10 (C2), 132.50 (C8), 131.50 (C12), 130.01 (C7), 128.50 (C13), 123.50 (C1), 121.50 (C11), 121.20 (C6); Anal. Calculated/Found: C:66.66/66.64, H:3.62/3.58, N:11.43/11.40, O: 18.28/18.26

EBL2: Red solid; Yield: 79%; M.P.: 202-204°C; FTIR (ATR, cm-1): 3069 ($C-H_{arom}$), 1702 (C=O), 1644 ($C=N_{im}$), 1575 ($C=N_{trz}$), 1498, 1447, 1418 ($C-C_{arom}$), 1382 (COO^-), 1296 ($C-O_{phen}$); ^1H NMR δ (ppm): 11.16 (s, 8H), 8.69 (s, 4H), 8.51 (s, 4H), 8.02 (s, 8H), 7.65 (d, 8H), 7.01 (d, 8H), 6.56 (d, 4H); ^{13}C NMR δ (ppm): 185.00 (C4), 176.50 (C3), 168.80 (C14), 160.00 (C9), 154.90 (C10), 156.00 (C5), 149.90 (C2), 133.50 (C8), 131.50 (C12), 129.90 (C7), 129.50 (C13), 123.90 (C1), 129.00 (C11), 120.90 (C6); Calculated/Found Analysis: C: 61.72/61.69 H: 3.17/3.16, N: 10.00/9.98, O: 25.12/25.10.

EBL3: Yellow, solid; Yield: 70%; M.P.: 330-332 °C; FTIR (ATR, cm^{-1}): 3286 ($N-H_{str}$), 1688 ($C=O_{str}$), 1651 ($C=N_{im}$), 1625 ($N-H_{str}$), 1557 ($C=N_{trz}$), 1496, 1409 ($C-C_{arom}$), 1361 (COO-), 1315 ($C-O_{phen}$); ^1H NMR δ (ppm): 11.17 (s, 4H), 8.69 (s, 4H), 7.98 (d, 8H), 7.76 (d, 8H), 7.49 (d, 8H), 6.96 (d, 4H), 6.84 (d, 8H), 4.89 (s, 2H); ^{13}C NMR δ (ppm): 169.50 (C4), 172.00 (C3), 169.00 (C14), 160.00 (C9), 157.50 (C10), 155.80 (C5), 132.60 (C8), 131.50 (C12), 130.02 (C7), 129.00 (C2), 128.20 (C13), 121.40 (C11), 121.20 (C6), 118.50 (C1); Anal. Calculated/Found: C: 66.77/66.72, H: 3.79/3.77, N: 13.74/13.72, O: 15.70/15.67.

EBL4: Yellow, solid; Yield: 74%; M.P.: 363°C (dec.); FTIR (ATR, cm^{-1}): 1693 ($C=O_{acid}$), 1567 ($C=N_{trz-broad}$), 1504, 1410 ($C-C_{arom}$), 1359 (COO^-), 1297 ($C-O_{phen}$); ^1H NMR δ (ppm): 11.19 (s, 8H), 8.68 (s, 4H), 8.52 (s, 4H), 8.01 (s, 8H), 7.61 (d, 8H), 6.98 (d, 4H), 6.85 (d, 8H), 4.85 (s, 2H); ^{13}C NMR δ (ppm): 179.00 (C4), 172.50 (C3), 168.90 (C14), 160.00 (C9), 153.80 (C10), 156.00 (C5), 132.50 (C8), 131.40 (C12), 130.70 (C7), 129.10 (C2), 129.50 (C13), 128.90 (C11), 120.90 (C6), 118.50 (C1); Anal. Calculated/Found: C: 61.81/61.79, H: 3.31/3.28, N: 12.01/12.00, O: 22.87/22.85.

The General synthesis of $[Cr/FeSalen/Saloph)_4]$ capped complexes (EBC1-EBC16) of target ligands

EBL1, EBL2, EBL3 or EBL4 (0.75 mmol) and $[(MSalen/Saloph)_2O]$ (M: Cr^{3+} or Fe^{3+}) [1.50 mmol (for EBL1 and EBL3) or 3.00 mmol (for EBL2 and EBL4)] were weighed and dissolved in 50 mL of methanol. The mixture was stirred under reflux for 3 hours at 75°C. Then the solvent was evaporated, and the solid product was recrystallized from methanol.

EBC1: Green solid; Yield: 70%; M.P.: 246-248 °C (dec.); μ_{eff} (B.M.): 3.83, FTIR (ATR, cm^{-1}): 3050, 3022, 3015 ($C-H_{arom}$), 2899 ($C-C_{aliph}$), 1696 (C=O), 1617 ($C=N_{imine}$), 1597 ($C=N_{trz}$), 1530,1458, 1440 ($C-C_{arom}$), 1380 (COO^-), 1290 ($C-O_{phen}$); Calculated/Found Analysis: C: 63.56/63.55, H: 3.88/3.85, Cr: 8.34/8.32, N: 10.11/10.09, O: 14.11/14.09.

EBC2: Green solid; Yield: 72%; M.P.: 244-247 °C (dec.); μ_{eff} (B.M.): 3.85, FTIR (ATR, cm^{-1}): 3045, 3020, 3010 ($C-H_{arom}$), 1699 (C=O), 1626 ($C=N_{im}$), 1592 ($C=N_{trz}$), 1532,1461,1440 ($C-C_{arom}$), 1380 (COO^-), 1297 ($C-O_{phen}$); Calculated/Found Analysis: C: 66.17/66.15, H: 3.60/358, N: 9.38/9.36, O: 13.10/13.07, Cr: 7.74/7.73,

EBC3: Brown solid; Yield: 77%; M.P.: 247-249 °C (dec.); μ_{eff} (B.M.): 1.72, FTIR (ATR, cm^{-1}): 3051,3027,3013 ($C-C_{arom}$), 2896 ($C-C_{aliph}$), 1698 (C=O), 1621 ($C=N_{im}$), 1595 ($C=N_{trz}$), 1537, 1465, 1440 ($C-C_{arom}$), 1382 (COO^-), 1296 ($C-O_{phen}$); Calculated /Found Analysis: C: 63.17/63.15, H: 3.86/3.82, N: 10.05/10.03, O: 14.02/14.01, Fe: 8.90/8.86.

EBC4: Brown solid; Yield: 76%; M.P.: 246-248 °C (dec.); μ_{eff} (B.M.): 1.71, FTIR (ATR, cm^{-1}): 3049, 3035, 3020 ($C-H_{arom}$), 1695 (C=O), 1618 ($C=N_{im}$), 1597 ($C=N_{trz}$), 1530, 1462, 1445 ($C-C_{arom}$), 1380

(COO^-), 1292 ($C-O_{phen}$); Calculated /Found Analysis: C: 65.79/65.77, H: 3.58/3.55, N: 9.33/9.31, O: 13.03/13.01, Fe: 8.27/8.25.

EBC5: Green solid; Yield: 80%; M.P.: 346°C (dec.); μ_{eff} (B.M.): 3.84, FTIR (ATR, cm^{-1}): 3045, 3021, 3012 ($C-H_{arom}$), 2895 ($C-H_{aliph}$), 1721, 1702 (C=O), 1624 (C=Nim), 1596 ($C=N_{trz}$), 1535, 1462, 1440 ($C-C_{arom}$), 1385 (COO^-), 1291 ($C-O_{phen}$); Analytical Calculated/Found: C: 60.98/60.96, H: 3.79/3.77, N: 9.24/9.22, O: 15.43/15.40, Cr: 10.56/10.53.

EBC6: Green solid; Yield: 74%; M.P.: 341°C (dec.); μ_{eff} (B.M.): 3.83, FTIR (ATR, cm^{-1}): 3041, 3025, 3013 ($C-H_{arom}$), 1721, 1704 (C=O), 1622 ($C=N_{im}$), 1597 ($C=N_{trz}$), 1533, 1466, 1441 ($C-C_{arom}$), 1388 (COO^-), 1295 ($C-O_{phen}$); Analytical Calculated/Found: C: 64.45/64.43, H: 3.45/3.42, N: 8.42/8.41, O: 14.06/14.03, Cr: 9.62/9.60.

EBC7: Dark Brown solid; Yield: 81%; M.P.: >300°C (dec.); μ_{eff} (B.M.): 1.70, FTIR (ATR, cm^{-1}): 3047, 3027, 3015 ($C-H_{arom}$), 2896 ($C-H_{aliph}$), 1722, 1700 (C=O), 1621 ($C=N_{im}$), 1595 ($C=N_{trz}$), 1537, 1465, 1441 ($C-C_{arom}$), 1382 (COO^-), 1294 ($C-O_{phen}$); Analytical Calculated/Found: C: 60.50/60.45, H: 3.76/3.74, N: 9.17/9.16, O: 15.31/15.29, Fe: 11.25/11.23.

EBC8: Red solid; Yield: 78%; M.P.: 290°C (dec.); μ_{eff} (B.M.): 1.71, FTIR (ATR, cm^{-1}): 3078, 3045, 3011 ($C-H_{arom}$), 1721 (C=O), 1602 ($C=N_{im}$), 1577 ($C=N_{trz}$), 1531, 1487, 1459, 1443, 1435 ($C-C_{arom}$), 1376, (COO^-), 1311 ($C-O_{phen}$); Analytical Calculated/Found: C: 63.99/63.96, H: 3.43/3.39, N: 8.36/8.34, O: 13.96/13.95, Fe: 10.26/10.23.

EBC9: Dark green solid; Yield: 79%; M.P.: 358 °C (dec.); μ_{eff} (B.M.): 3.86, FTIR (ATR, cm^{-1}): 3011 ($C-H_{arom}$), 1685 ($C=O_{acid}$), 1620 ($C=N_{im}$), 1594 ($N-H_{bend}$), 1565 ($C=N_{trz}$), 1542, 1496, 1470, 1441 ($C-C_{arom}$), 1385 (COO^-), 1286 ($C-O_{phen}$); Calculated/Found Analysis: C: 63.61/63.59, H: 3.96/3.93, N: 11.24/11.21, O: 12.84/12.83, Cr: 8.34/8.32.

EBC10: Dark green solid; Yield: 75%; M.P.: 352 °C (dec.); μ_{eff} (B.M.): 3.84, FTIR (ATR, cm^{-1}): 3016 ($C-H_{arom}$), 1688 ($C=O_{acid}$), 1620 ($C=N_{im}$), 1596 ($N-H_{bend}$), 1565 ($C=N_{trz}$), 1542, 1496, 1470, 1442 ($C-C_{arom}$), 1385 (COO^-), 1290 ($C-O_{phen}$); Calculated/Found Analysis: C: 66.22/66.20, H: 3.68/3.65, N: 10.44/10.43, O: 11.92/11.90, Cr: 7.75/7.73.

EBC11: Brown solid; Yield: 82%; M.P.: 250 °C (dec.); μ_{eff} (B.M.): 1.72, FTIR (ATR, cm^{-1}): 3014 ($C-H_{arom}$), 1687 ($C=O_{acid}$), 1619 ($C=N_{im}$), 1597 ($N-H_{bend}$), 1564 ($C=N_{trz}$), 1543, 1495, 1469, 1443 ($C-C_{arom}$), 1386 (COO^-), 1289 ($C-O_{phen}$); Calculated/Found Analysis: C: 63.22/63.19, H: 3.94/3.93, N: 11.17/11.13, O: 12.76/12.74, Fe: 8.91/8.89.

EBC12: Brown solid; Yield: 80%; M.P.: 262 °C (dec.); μ_{eff} (B.M.): 1.73, FTIR (ATR, cm^{-1}): 3416 ($N-H_{str}$), 3288 ($C-H_{arom}$), 3045, 3013 ($C-H_{aliph}$), 1688 ($C=O_{acid}$), 1602 ($C=N_{im}$), 1575 ($C=N_{trz}$), 1532, 1493, 1459, 1435 ($C-C_{arom}$), 1376 (COO^-), 1312 ($C-O_{phen}$); Calculated/Found Analysis: C: 65.84/65.82, H: 3.66/3.63, N: 10.38/10.36, O: 11.85/11.82, Fe: 8.27/8.26.

EBC13: Green solid; Yield: 79%; M.P.: 350°C (dec.); μ_{eff} (B.M.): 3.82; FTIR (ATR, cm^{-1}): 3386 ($N-H_{str}$), 3050, 3021 ($C-H_{arom}$), 2915, 2848 ($C-H_{aliph}$), 1696 ($C=O_{acid}$), 1616 (C=N im), 1596 ($N-H_{bend}$), 1568 ($C=N_{trz}$), 1541, 1503, 1467, 1440 ($C-C_{arom}$), 1358 (COO^-), 1287 (C-Ophen); Analytical Calculated/Found: C: 61.01/61.00, H: 3.84/3.82, N: 9.96/9.93, O: 14.63/14.61, Cr: 10.56/10.53.

EBC14: Green solid; Yield: 71%; M.P.: 354°C (dec.); μ_{eff} (B.M.): 3.84; FTIR (ATR, cm^{-1}): 3396 ($N-H_{str}$), 3017 ($C-H_{arom}$), 1699 ($C=O_{acid}$), 1620 ($C=N_{im}$), 1600 ($N-H_{bend}$), 1574 ($C=N_{trz}$), 1544, 1508, 1468, 1440 ($C-C_{arom}$), 1388 (COO^-), 1292 ($C-O_{phen}$); Analytical Calculated/Found: C: 64.48/64.45, H: 3.50/3.47, N: 9.07/9.04, O: 13.33/13.32, Cr: 9.62/9.60.

EBC15: Dark brown solid; Yield: 76%; M.P.: 243°C (dec.); μ_{eff} (B.M.): 1.72; FTIR (ATR, cm^{-1}): 3388

(N-H_{str}), 3051, 3023 (C-H_{arom}), 2917, 2851 (C-H_{aliph}), 1698 (C=O_{acid}), 1618 (C=N_{im}), 1597 (N-H_{bend}), 1568 (C=N_{trz}), 1542, 1504, 1468, 1443 (C-C_{arom}), 1360 (COO^-), 1288 (C-O_{phen}); Analytical Calculated/Found: C: 60.53/60.51, H: 3.81/3.80, N: 9.88/9.87, O: 14.51/14.50, Fe: 11.26/11.23.

EBC16: Dark brown solid; Yield: 77%; M.P.: 250°C (dec.); μ_{eff} (B.M.): 1.73; FTIR (ATR, cm^{-1}): 3394 (N-H_{str}), 3019 (C-H_{arom}), 1698 (C=O_{acid}), 1618 (C=N_{im}), 1598 (N-H_{bend}), 1572 (C=N_{trz}), 1541, 1505, 1466, 1443 (C-C_{arom}), 1385 (COO^-), 1290 (C-O_{phen}); Analytical Calculated/Found: C: 64.02/64.00, H: 3.47/3.45, N: 9.01/9.00, O: 13.23/13.21, Fe: 10.26/10.23.

RESULTS AND DISCUSSION

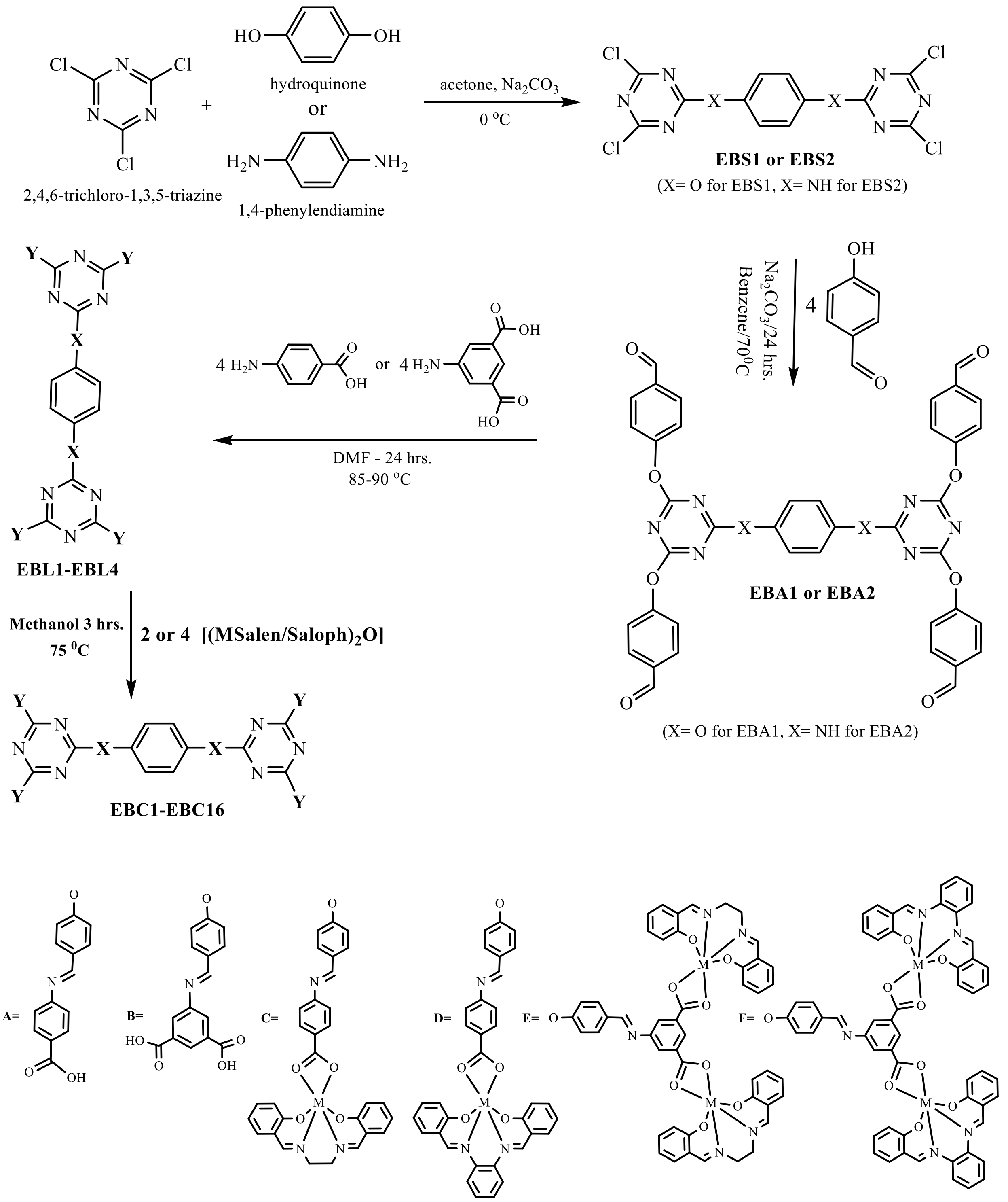
This study focuses on synthesizing ligands based on cyanuric chloride (CC) and employing hydroquinone (HQ) and p-phenylenediamine (PFDA) as central units. The reactions between **HQ** and **CC**, as well as **PFDA** and **CC**, resulted in the formation of specific compounds, **EBS1** and **EBS2**, respectively. These compounds were further reacted with p-hydroxybenzaldehyde (PHBA) to produce EBA1 and EBA2. Subsequent reactions of **EBA1** with p-aminobenzoic acid (PABAc) and 5-aminoisophthalic acid (5AIPA) yielded target ligands **EBL1** and **EBL2**, while similar reactions with **EBA2** produced **EBL3** and **EBL4**, respectively. The structures of these ligands were characterized using various analytical and spectroscopic techniques. Additionally, reactions with $[Cr/Fe(Salen/Saloph)]_2O$ initial complexes yielded capped complexes, with the structures determined through analytical and spectroscopic methods (**Scheme 1**).

Interpretation of ^1H NMR and ^{13}C NMR spectra

When examining the ^1H NMR spectra of the intermediate compound coded as **EBS1** and **EBS2**, the singlet signal observed at 4.85 ppm for an aromatic-NH group for **EBS2**, and the singlet signal observed at 6.56 ppm and 7.61 ppm confirms the successful synthesis of this compound, respectively. Similarly, upon inspection of the ^{13}C NMR spectrum of these compounds, signals corresponding to two carbons were observed at 187.0 ppm, four carbons were observed at 170.0 ppm, and in the aromatic region, signals were observed at 150.4 ppm for two carbons, and at 115.8 ppm for four carbons for **EBS1** and 169.0 ppm, four carbons were observed at 163.8 ppm, and in the aromatic region, signals were observed at 133.9 ppm for two carbons, and at 122.3 ppm for four carbons for **EBS1**, further substantiate the successful synthesis of this compound (**Figure 1** and **Figure 2**), (**Figure S1, S2, S9, and S10**, Supporting information) [15-17].

When we examined the ^1H NMR spectrum of the intermediate products labeled as **EBA1** and **EBA2**, singlets (4H) observed at 9.87 ppm for the HC=O group, doublets (8H) at 7.84-7.85 ppm, doublets (8H) at 7.15 ppm, and doublet signals (4H) at 7.07-6.40 ppm, have successfully proven the synthesis of these compounds. Upon examining the ^{13}C NMR spectra of these compounds, signals at 192.1 ppm for 4 carbon C_9, at 178.9-184.9 ppm for 4 carbons C_4, at 171.9-177.5 ppm for 2 carbons C_3, at 161.1-161.2 ppm for 4 carbons C_5, at 128.8/150.01 ppm for 2 carbons C_2, at 131.5-132.5 ppm for 8 carbons C_7, at 118.7-123.3 ppm for 4 carbons C_1, and at 121.8-122.0 ppm for 8 carbons C_6, as well as signals at 116.1/133.2 ppm for 4 carbons C_8, provide further evidence of the successful synthesis of these compounds (Figure 3 and Figure 4), (Figure S3, S4, S11, and S12, Supporting information) [18, 19].

Analysis of the ^1H NMR spectrum revealed that ligands labeled **EBL1** and **EBL3**, weak signals observed at 10.60-11.17 ppm correspond to acidic protons, singlets observed at 8.69 ppm correspond to four hydrogens (HC=N), doublets (8H) at 7.98-8.24 ppm, doublets (8H) at 7.76-7.79 ppm, doublets (8H) at 7.49-7.60 ppm, doublets (8H) at 6.96-7.08 ppm, and doublets (4H) at 6.84-7.01 ppm. These signals successfully prove the synthesis of these compounds.



Comp.	**X**	**Y**	**Comp.**	**X**	**Y**	**M**	**Comp.**	**X**	**Y**	**M**	**Comp.**	**X**	**Y**	**M**	**Comp.**	**X**	**Y**	**M**
EBL1	O	A	**EBC1**	O	C	Cr(III)	**EBC5**	O	E	Cr(III)	**EBC9**	NH	C	Cr(III)	**EBC13**	NH	E	Cr(III)
EBL2	O	B	**EBC2**	O	D	Cr(III)	**EBC6**	O	F	Cr(III)	**EBC10**	NH	D	Cr(III)	**EBC14**	NH	F	Cr(III)
EBL3	NH	A	**EBC3**	O	C	Fe(III)	**EBC7**	O	E	Fe(III)	**EBC11**	NH	C	Fe(III)	**EBC15**	NH	E	Fe(III)
EBL4	NH	B	**EBC4**	O	D	Fe(III)	**EBC8**	O	F	Fe(III)	**EBC12**	NH	D	Fe(III)	**EBC16**	NH	F	Fe(III)

Scheme 1

Synthetic route of ligand and complexes

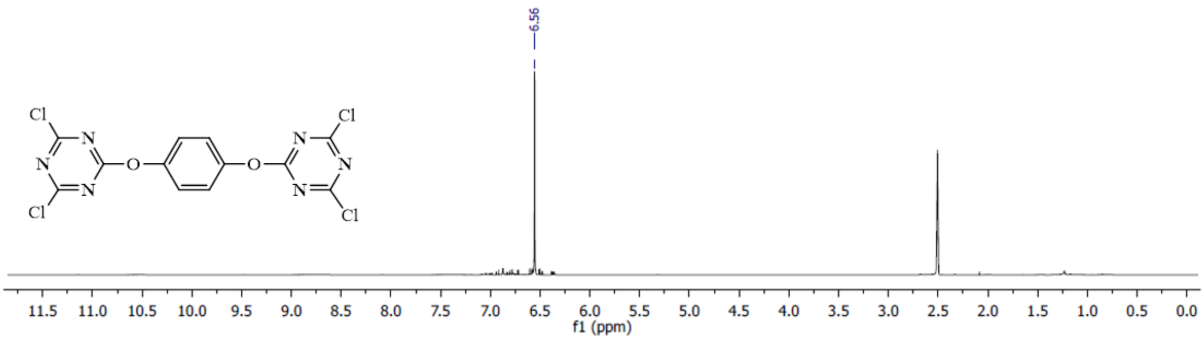


Figure 1

^1H NMR spectrum of EBS1.

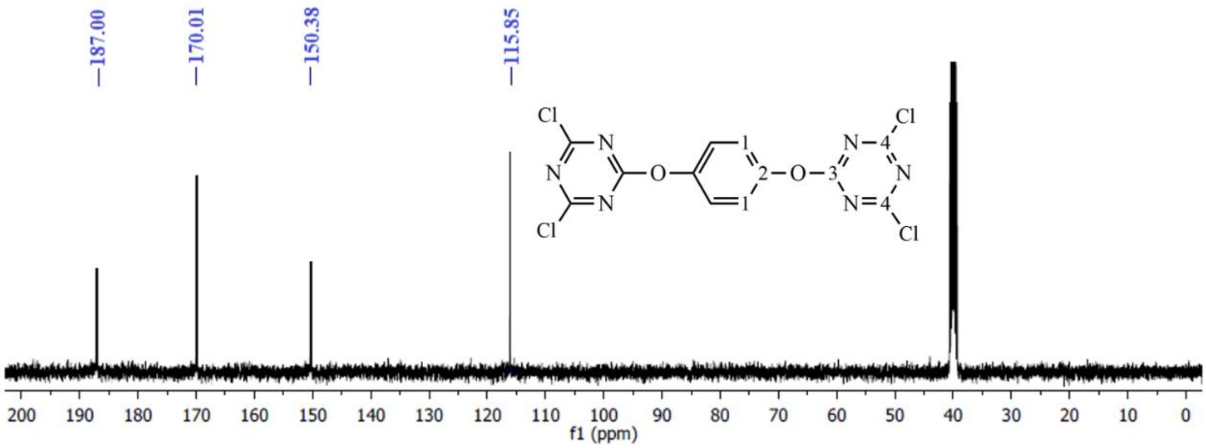


Figure 2

^{13}C NMR spectrum of EBS1

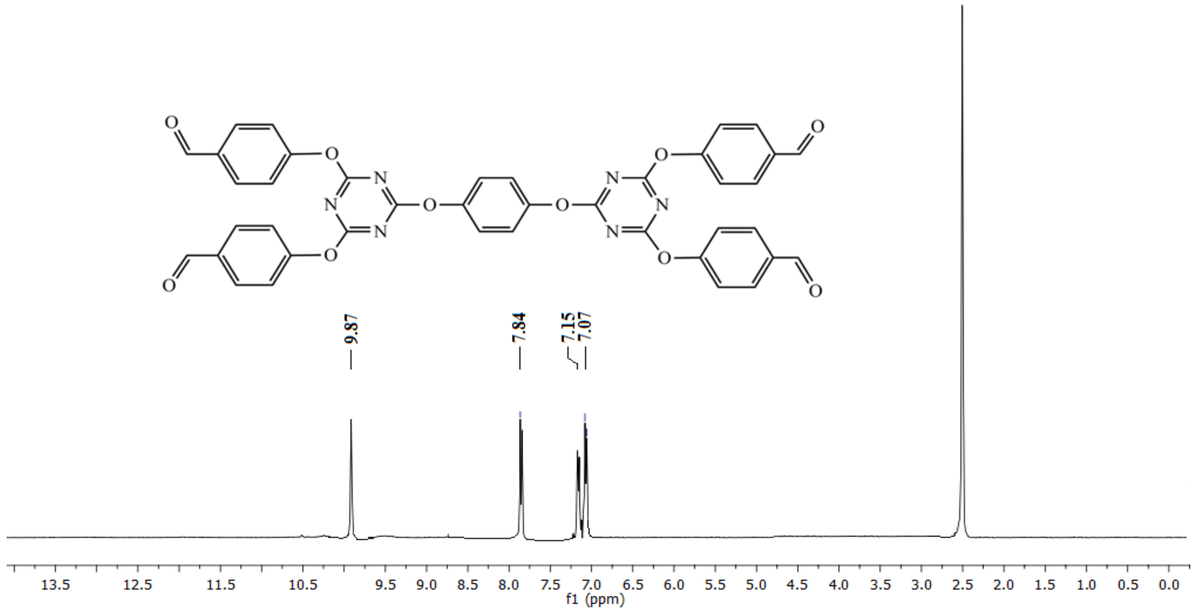


Figure 3

^1H NMR spectrum of EBA1

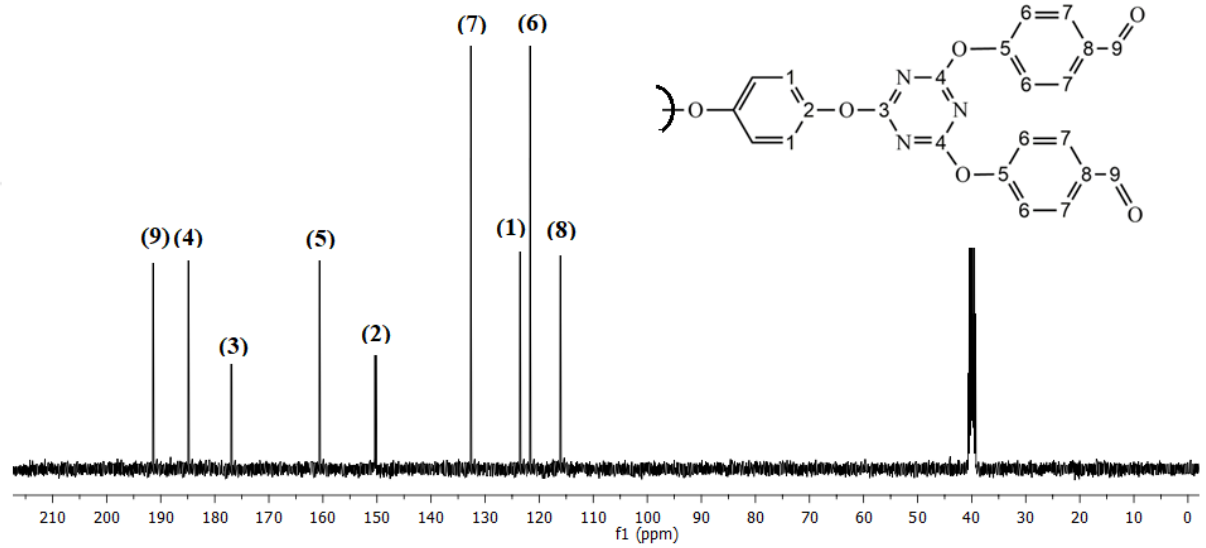


Figure 4
^{13}C NMR spectrum of EBA1

Similarly, when we examine the ^{1}H NMR spectrum of ligands labeled **EBL2** and **EBL4**, weak signals observed at 11.16-11.19 ppm correspond to acidic protons, singlets observed at 8.68-8.69 ppm correspond to four hydrogens (HC=N), singlets (4H) at 8.51-8.52 ppm, singlets (8H) at 8.01-8.02 ppm, doublets (8H) at 7.61-7.65 ppm, doublets (8H) at 6.98-7.01 ppm, doublets (8H) at 6.56-6.84 ppm, and a weak singlet signal (2H) observed at 4.85 ppm in the spectrum of **EBL4**. These signals also prove the successful synthesis of these compounds. Additionally, ^{13}C NMR signals were observed at 184.9/169.5 ppm for 4 carbons C_4, at 172.0-176.2 ppm for 2 carbons C_3, at 168.9-179.5 ppm for 4 carbons C_{14}, at 160.0 ppm for 4 carbons C_9, at 157.5 ppm for 4 carbons C_{10}, at 155.8-155.9 ppm for 4 carbons C_5, 149.1 and 129.0 ppm for 2 carbons C_2, at 132.5-132.6 ppm for 4 carbons C_8, at 131.5 ppm for 8 carbons C_{12}, at 130.0 ppm for 8 carbons C_7, at 128.2-128.5 ppm for 4 carbons C_{13}, at 123.5 and 118.5 ppm for 4 carbons C_1, at 121.4-121.5 ppm for 8 carbons C_{11}, and at 121.2 ppm for 8 carbons C_6 also serve as evidence for the successful synthesis of these compounds, respectively. To examine the ^{13}C NMR spectrum of **EBL2** and **EBL4** codded ligands, signals observed at 185.0 and 179.0 ppm for 4 carbons C_4, at 172.5-176.5 ppm for 2 carbons C_3, at 168.8-168.9 ppm for 8 carbons C_{14}, at 160.0 ppm for 4 carbons C_9, at 153.8-156.0 ppm for 4 carbons C_{10}, at 154.9-156.0 ppm for 4 carbons C_5, at 140.9 and 129.1 ppm for 2 carbons C_2, at 132.5-133.5 ppm for 4 carbons C_8, at 131.4-131.5 ppm for 8 carbons C_{12}, at 139.9 and 130.7 ppm for 8 carbons C_7, at 129.5 ppm for 4 carbons C_{13}, at 129.0 and 118.5 ppm for 4 carbons C_1, at 123.9 and 128.9 ppm for 8 carbons C_{11}, and at 120.9 ppm for 8 carbons C_6 further confirm the successful synthesis of these compounds (Figure 5 and Figure 6), (Figure S5, S6, S7, S8, S13, S14, S15, and S16, Supporting information) [20].

Interpretation of FTIR spectra

In our study, FTIR spectra were obtained to determine the functional groups of the intermediate compounds, target ligands, and target complexes synthesized. Accordingly, when examining the FTIR spectra of the intermediate products coded as **EBS1** and **EBS2**, stretching bands corresponding to s-triazine rings were observed at 1551 cm^{-1} and 1539 cm^{-1}, respectively. Additionally, in the spectrum of **EBS2**, stretching bands for N-H at 3375 cm^{-1} and 3362 cm^{-1}, bending bands for N-H at 1605 cm^{-1}, and stretching bands for C-Car in the aromatic region (1504 cm^{-1}, 1500 cm^{-1}, and 1431 cm^{-1}) were observed. These observations, as illustrated in **Figure S17 and Figure S18**, confirm the successful synthesis of these compounds. When examining the FTIR spectra of the intermediate products coded as EBA1 and EBA2, the observation of C=O stretching bands at 1698 and 1694 cm^{-1}, respectively, stretching bands of s-triazine rings at 1585 cm^{-1} and 1562 cm^{-1}, and specific bands in the aromatic region proved that the compounds were successfully synthesized (**Figure S19 and Figure S20,** Supplementary Information**)**.

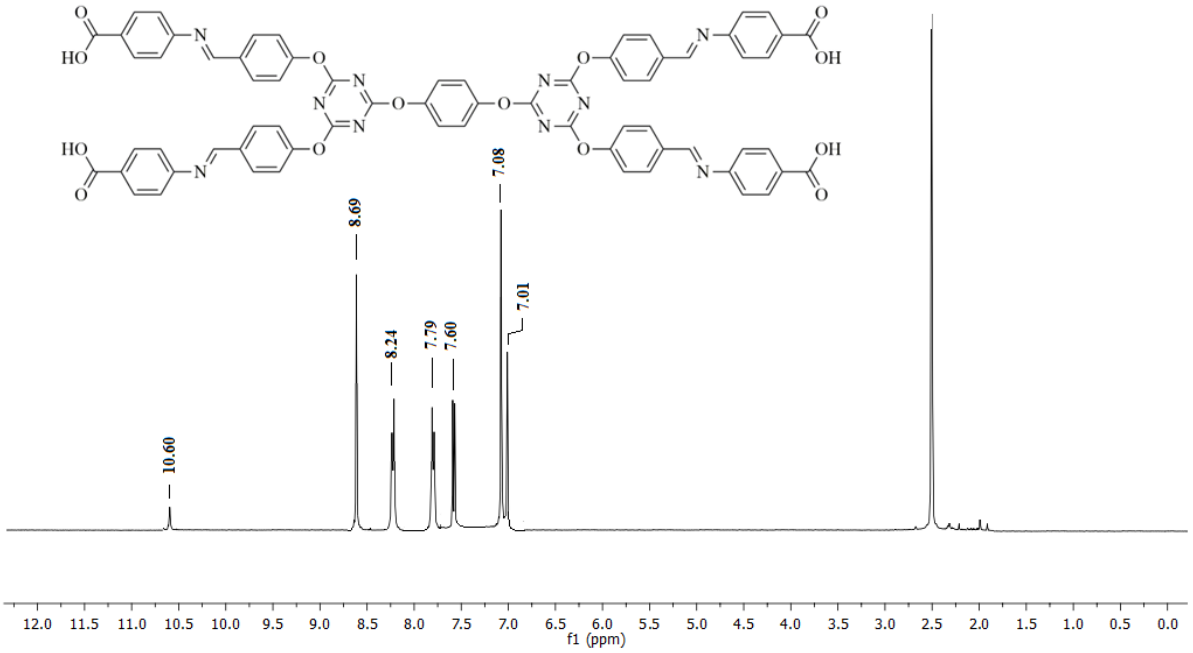


Figure 5

^{1}H NMR spectrum of EBL1

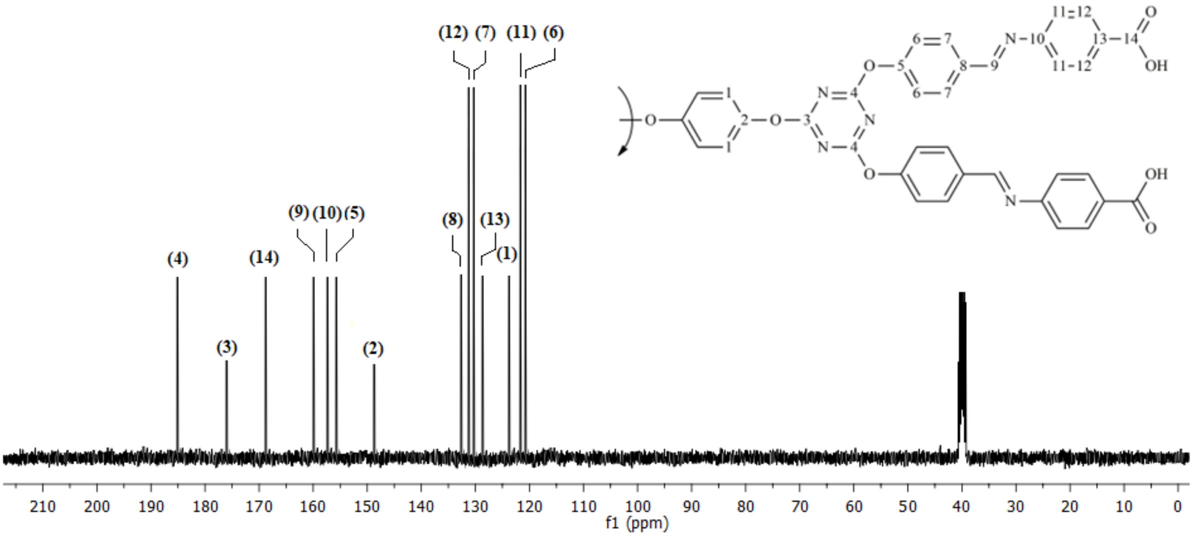


Figure 6

^{13}C NMR spectrum of EBL1

When we examine the FTIR spectra of **EBL1**, **EBL2**, **EBL3,** and **EBL4** target ligands, the C=O stretching bands observed at 1698 and 1694 cm^{-1} in the previous compounds disappear, and instead of them, C=O$_{acid}$ stretching bands appear in the range of 1682-1702 cm^{-1}, and COOH bending bands appear in the range of 1359-1382 cm^{-1} proved that these compounds were successfully synthesized (**Figure S21-Figure S24,** Supplementary Information**)**. When we examine the FTIR spectra of the target complexes coded **EBC1-EBC16**, that the C=O$_{acid}$ stretching bands observed in the range of 1682-1702 cm^{-1} for the ligands shift to higher frequencies (1682-1721 cm^{-1}), and the COO$^-$ bending bands in the range of 1359-1382 cm^{-1} shift to higher frequencies (1358-1388 cm^{-1}) showed that these complexes were synthesized, successfully (**Table S1, Table S2** and **Figure S25-Figure S31,** Supplementary

Information**)**. While the FTIR spectra of all synthesized ligands as well as complexes are attached to supplementary information [17, 21-23].

Evaluation of TGA results of some complexes

Thermal gravimetric analysis (TGA) was conducted on certain complexes obtained in our study, namely **EBC3**, **EBC7**, **EBC11** and **EBL15**, to determine their thermal stabilities. The TGA analyses were performed under an N_2 atmosphere with a heating rate of 10 °C/min in the temperature range of 50-800 °C. **Figure 7**, below shows the TGA graph of the **EBC3** complex, while TGA graphs of the other complexes are attached to the supplementary information.

Examination of the TGA plot of the complexes **EBC3**, **EBC7**, **EBC11,** and **EBC15** revealed three stages of decomposition. In the first stage, mass losses of 6.75-8.75 % occurred as the CO_2 group detached from the molecule in the range of 125-205 °C, resulting in the detachment of the FeSalen caps from the main structure. Theoretical mass losses at this stage were calculated as 7.01-8.87 %. In the second stage, mass losses of 21.97-35.10 % occurred as the organic structure attached to the cyanuric chloride centers, excluding the Fe(Salen/Saloph) caps, separated from the structure in the range of 290-440 °C. Theoretical mass loss at this stage was 22.24-35.42%. In the final stage, mass losses of 34.22-46.35% indicated the decomposition of the salen caps in the range of 525-640 °C, with a theoretical mass loss of 37.34-47.23%. Since the heating process was carried out up to 800 °C, no further mass losses were observed beyond this point. FTIR and ICP-AES analyses of the remaining materials after TGA revealed that the residual materials were cyanuric chloride centers and Fe_2O_3 resulting from decomposition. Theoretical data are in good harmony with the experimental data, and consistent with other spectroscopic data (**Figure S32-Figure S35,** Supplementary Information**)** [17, 21-26].

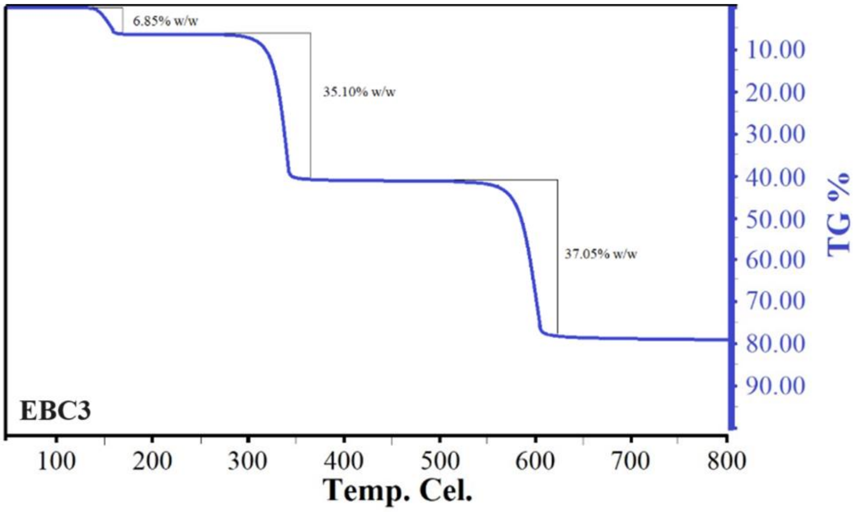


Figure 7
TGA graph of EBC3

Interpretation of magnetic susceptibility measurements

In our study, magnetic susceptibility values of the complexes synthesized were measured at room temperature using a Gouy balance. The molar magnetic susceptibility (μeff) values of all complexes in terms of Bohr magneton (B.M.) are provided in experimental detail.

All synthesized ligands, **EBC1**, **EBC2**, **EBC5**, **EBC6**, **EBC9**, **EBC1**0, **EBC13** and **EBC14** coded complexes, possessing a d^3 electron configuration, were measured paramagnetically. Each Cr^{3+} ion exhibited values of 3.83, 3.85, 3.84, 3.83, 3.86, 3.84, 3.82, and 3.84 B.M. respectively, with a $t_{2g}{}^3e_g{}^0$ electron configuration. Similarly, complexes coded as **EBC3**, **EBC4**, **EBC7**, **EBC8**, **EBC11**, **EBC12**, **EBC15** and **EBC16**, possessing a d^5 electron configuration, were measured paramagnetically. Each Fe^{3+}

ion exhibited values of 1.72, 1.71, 1.70, 1.71, 1.72, 1.73, 1.72, and 1.73 B.M. respectively, with a low spin $t_{2g}^5e_g^0$ electron configuration. Literature review indicates that [Fe(Salen/Saloph)$_2$O] complexes possess a $t_{2g}^3e_g^2$ electron configuration and are high-spin and five-coordinate complexes. These complexes, capable of coordinating with another ligand, are termed ligand complexes. When these ligand complexes coordinate with another monodentate ligand, their coordination numbers remain unchanged, and their electronic configurations remain the same. However, when they coordinate with a bidentate ligand using both of its arms, they transition to a low-spin hexacoordinate electron configuration. Considering all the complexes synthesized in our study, denoted as [Cr/Fe(Salen/Saloph)], were measured as low-spin, we evaluate their geometries as distorted octahedral [17, 23, 27-30].

CONCLUSIONS

In this work, four new original ligands (EBL1-EBL4) were synthesized. These ligands were characterized by using elemental analysis, FTIR, ^{1}H and ^{13}C NMR, and ICP-AES spectroscopies. The [MSalen/Saloph] (M = Cr^{3+}, Fe^{3+}) capped complexes were obtained from the reaction of these ligands and ligand complexes ([M(Salen/Saloph)]$_2$O). The structure of these complexes determined as paramagnetic was characterized by using elemental analysis, Magnetic Susceptibility, FTIR, and TGA methods. Complexes (EBC1, EBC2, EBC5, EBC6, EBC9, EBC10, EBC13 and EBC14) showed good harmony with the d^3 (S = 3/2) Cr^{3+} ions, and the other complexes (EBC3, EBC4, EBC7, EBC8, EBC11, EBC12, EBC15 and EBC16) showed good harmony with the low spin d^5 (S = 1/2) Fe^{3+} ions. That is, all Cr^{3+} and Fe^{3+} complexes are in distorted octahedral structure with $t_{2g}^3e_g^0$ and $t_{2g}^5e_g^0$ electronic configuration, respectively. The findings indicated that the carboxylate groups were coordinated to the metal center through both oxygen atoms. Thermogravimetric analysis was performed on selected complexes coded as EBC3, EBC7, EBC11, and EBC15, revealing that they possessed high thermal stability, making them suitable for use in chemical reactions and other applications.

Ethical Statement

This study is derived from a master's thesis entitled “s-Triazin kullanılarak çok dişli Schiff bazlarının sentezi, karakterizasyonu ve bazı geçiş metali komplekslerinin incelenmesi”, submitted under the supervision of Prof. Dr. Şaban UYSAL on 24/08/2023 date.

Author Contributions

Research Design (CRediT 1) E.B. (%40) - A.H.M. (%30) - Ş.U. (%30)
Data Collection (CRediT 2) E.B. (%45) - A.H.M. (%30) - Ş.U. (%25)
Research - Data Analysis - Validation (CRediT 3-4-6-11) E.B. (%40) - A.H.M. (%30) - Ş.U. (%30)
Writing the Article (CRediT 12-13) E.B. (%20) - A.H.M. (%50) - Ş.U. (%30)
Revision and Improvement of the Text (CRediT 14) E.B. (%20) - A.H.M. (%50) - Ş.U. (%30)

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Conflict of interest

The authors declare no conflict of interest for the present study.

Data availability

Data will be made available on request.

Sustainable Development Goals (SDG)

Sustainable Development Goals: Not supported.

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