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Research Article

The synthesis of polymeric s-triazine Schiff bases and investigation of [(Fe(III)/Mn(III)(Salen)Cl] metal complexes

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ABSTRACT

In this study, 2,4,6-triamino-1,3,5-triazine (melamine) as the starting material. A one-way Schiff base reaction took place with the condensation reaction of melamine and p-hydroxybenzaldehyde. Then, monopodal s-triazine-centered Schiff base ligand complexes were obtained by obtaining a single oxygen-bridged compound of [(Fe(III)/Mn(III)Salen)Cl] ligand complexes, which we synthesized monopodal Schiff base s-triazine monomer by the literature. The obtained unidirectional s-triazine-centered Schiff base ligand complexes were polymerized under reflux with different dialdehyde compounds. Consequently, the structures of the obtained all ligands and complexes were characterized using elemental analysis, FT-IR spectroscopy, ^{1}H-NMR spectroscopy, ESI-LS-MS spectroscopy, TGA-DTA analysis and magnetic susceptibility measurement techniques.

Araştırma Makalesi

Polimerik s-triazin Schiff bazlarının sentezi ve [(Fe(III)/Mn(III)(Salen)Cl] metal komplekslerinin incelenmesi

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ÖZ

Bu çalışmada çıkış maddesi olarak 2,4,6-triamino-1,3,5-triazin (melamin) kullanıldı. Melamin ve p-hidroksibenzaldehitin kondensasyon reaksiyonu ile tek yönlü Schiff baz reaksiyonu gerçekleşti. Daha sonra, monopodal Schiff baz s-triazin monomeri literatüre uygun olarak sentezlenen [(Fe(III)/Mn(III)Salen)Cl] ligand komplekslerini tek oksijen ile köprülü bileşiği elde edilerek monopodal s-triazin merkezli Schiff baz ligand kompleksleri elde edildi. Elde edilen tek yönlü s-triazin merkezli Schiff baz ligand kompleksleri farklı dialdehit bileşikleri geri soğutucu altında polimerleştirildi. Sonuç olarak, elde edilen ligandların, monomerik ve polimerik komplekslerinin yapıları elementel analiz, FT-IR spektroskopisi, ^{1}H-NMR spektroskopisi, TGA-DTA analizi, viskozimetre ve manyetik sussebtibilite ölçüm teknikleri kullanılarak aydınlatıldı.

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1. Introduction

Melamine resins have been used in many applications, including manufacturing plastic dishes under the trade name Melmac. Melamine-based co-polymers have interesting applications in material science because of their optical, electrical, and optoelectronic properties (Wang and Zhang, 2004; Uysal, 2013). Melamine resins have high transparency and enormous thermal and mechanical stability Melamine, one of the s-triazine compounds, is rapidly increasing in use in polymer chemistry, coordination chemistry, environmental chemistry, biochemistry, dyestuff chemistry, pharmaceutical chemistry and the electronics industry (Wimmer et al., 1992; Uysal et al., 2012). In addition, s-triazine Schiff base compounds are used in medicine, especially as molecular magnetic materials and such heterocyclic compounds are used as active ingredients of antitumor and anticancer drugs (Koc and Uysal, 2016; Arslaner et al., 2017; Ozer et al., 2023). Melamine compounds have gained importance in environmental chemistry, metal-organic lattice structures and gas storage (Yu et al., 2008).

Melamine was used as the central s-triazine group in the synthesis of Schiff base ligands (Uysal and Koc, 2016). Since melamine has symmetrical three-way amine groups, Schiff base-containing melamine ligands were obtained by condensation reaction with different aldehyde groups (Koc and Uysal, 2016). Monopodal melamine-centered ligand monomer complexes were obtained by coordinating the obtained s-triazine monomer with the salen ligand complexes with a single oxygen (Celikbilek and Koc, 2014). Polymeric-s-triazine [(Fe(III)/Mn(III)Salen)] complexes were obtained by polymerizing these complexes with dialdehydes (terephthalaldehyde, glutaraldehyde, phtaldehyde and isophthalaldehyde) (Karipcin and Karatas, 2001; Uysal, 2013).

2. Experimental

2.1 Materials and methods

The chemicals were purchased from Aldrich and Merck was used as received. Melting points were measured using an Optimelt Automated Melting Point System (Digital Image Processing Technology) SRS apparatus (Nyköping-Sweden). Elemental analyses (C, H, N) were performed using a Leco, CHNS-932 model analyzer (Massachusetts, USA). ^{1}H NMR spectra were recorded by the Varian, 400 M spectrometer at room temperature. (California, USA). FT-IR spectra were recorded using a Perkin-Elmer Spectrum 100 with Universal ATR Polarization Accessory (Shelton, USA). Magnetic susceptibilities of the metal samples were measured at 296 K using a Sherwood Scientific MX Gouy magnetic susceptibility apparatus (Gouy method) with Hg[Co(SCN)4] as a calibration by the constant magnetic field. The effective magnetic moments, μeff, per metal atom were calculated from the expression, $\mu_{eff.} = 2.84\sqrt{\chi_M T}$ B.M., where χ_M is the molar susceptibility (Cambridge, UK). TGA analyses of the compounds were performed on the Mettler Toledo brand TGA/DSC-2 model Thermal Analysis System device

2.2. 4-((4,6-diamino-1,3,5-triazine-2-imino) methyl) phenol

Melamine was mixed with 50 mL of benzene solvent (1 mmol, 1.26 g). The resulting mixture was boiled under reflux with stirring for one hour with 4-hydroxybenzaldehyde (1 mmol, 1.22 g). It was slowly added to the mixture and stirred at 90 °C for 26 s under reflux. The mixture was filtered off to separate the precipitated compound. The precipitated white precipitate was dried in the oven. $C_{10}H_{10}N_6O$: ^{1}H NMR (DMSO-d_6, ppm): 9.76 (s, H, OH), 8.39 (s, H, CH=N), 7,74-7,72 (d, 2H, Ar-H), 6,89-6,91 (d, 2H, Ar-H), 6.11 (s, 4H, NH_2). FT-IR(cm^{-1}) 3467-3414 (NH_2), 3167 (OH), 1649 (C=N), 1526 (C=N_{tr}).

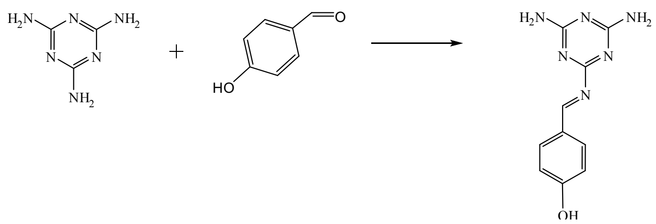

Figure 1. Monomer Schiff base ligand.

2.3. Synthesis of Salen ligand and Salen complexes

Synthesis of salen ligand and salen complexes were synthesized according to the cited literature. (Kopel et al., 1998; Gembicky et al., 2000).

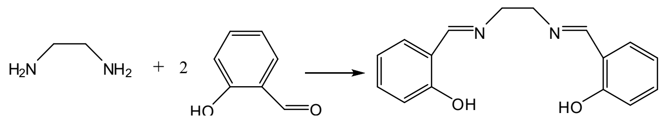

Figure 2. Salen ligand.

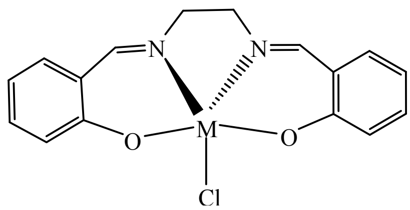

Figure 3. [M(salen)Cl] complexes. M = Fe(III), Mn(III)

2.4. Synthesis of 4-((4,6-diamino-1,3,5-triazine-2-imino)methyl)phenol [Fe(III)/Mn(III)(salen)Cl]

A suspension solution of the obtained monomer (1 mmol, 0.23 g) in ethanol was prepared. [Fe(III)/Cr(III)(salen)Cl] (1 mmol, 0.37 g, 1 mmol, 0.37 g) complex compound dissolved in ethanol was added onto the monomer. The reaction mixture was stirred at 100 °C for four hours under reflux. The reaction solution was filtered, and the precipitate was dried in the oven. $FeC_{27}H_{26}N_8O_3$: FT-IR(cm^{-1}) 3317-3354 (NH_2), 1635 (C=N), 1546 (C=$N_{triazin}$). $MnC_{27}H_{26}N_8O_3$: FT-IR(cm^{-1}) 3327-3357 (NH_2), 1625 (C=N), 1555 (C=$N_{triazin}$).

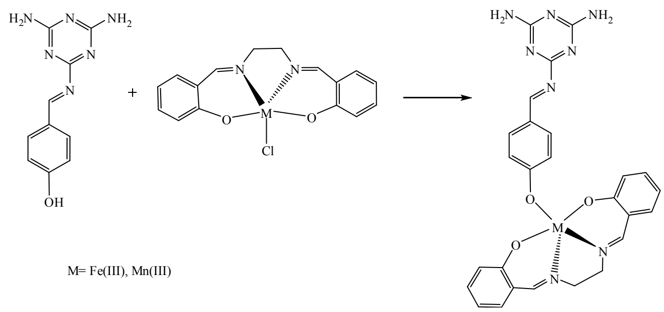

Figure 4. Ligand complexes.

2.5. Synthesis of 4-((4,6-diamino-1,3,5-triazine-2-imino)methyl)phenol [Fe(III)/Mn(III)(salen)Cl] polymer complexes

[Fe(III)/Mn(III)(salen)Cl] complex of 4-((4,6-diamino-1,3,5-triazine-2-imino)methyl)phenol (1 mmol, 0.57 g, 1 mmol, 0.56 g) was dissolved in 50 mL of acetonitrile and stirred under reflux for one hour. Terephthalaldehyde, glutaraldehyde, phthalaldehyde, isophthalaldehyde (1 mmol, 0.14 g/2 mL/2 mL/2 mL) were added to the resulting mixture, respectively. The mixture was stirred at 100°C for twenty-four hours and 5-6 drops of acetic acid catalyst was added. It was mixed for a while until the polymer formed and a color change was observed. The mixture was quickly filtered, and the precipitate was dried in the oven. $[TFAMHBAFe]_n$: FT-IR(cm^{-1}) 1687,1592 (C=N), 1549 ($C{=}N_{triazin}$). $[TFAMHBAMn]_n$: FT-IR(cm^{-1}) 1675, 1587 (C=N), 1540 ($C{=}N_{triazin}$). $[GTAMHBAFe]_n$: FT-IR(cm^{-1}) 3051, 3027, 2898 (CH2), 1623, 1597 (C=N), 1539 ($C{=}N_{triazin}$). $[GTAMHBAMn]_n$: FT-IR(cm^{-1}) 2924, 2898, 2851 (CH_2), 1623, 1596 (C=N), 1537 ($C{=}N_{triazin}$). $[FAMHBAFe]_n$ FT-IR(cm^{-1}) 1687, 1592 (C=N), 1549 ($C{=}N_{triazin}$). $[FAMHBAMn]_n$: FT-IR(cm^{-1}) 1675, 1587 (C=N), 1540 ($C{=}N_{triazin}$). $[FAMHBAFe]_n$ FT-IR(cm^{-1}) 1687, 1592 (C=N), 1549 ($C{=}N_{triazin}$). $[FAMHBAMn]_n$: FT-IR(cm^{-1}) 1675, 1587 (C=N), 1540 ($C{=}N_{triazin}$). $[IFAMHBAFe]_n$: FT-IR(cm^{-1}) 1624, 1597 (C=N), 1538 ($C{=}N_{triazin}$). $[IFAMHBAMn]_n$: FT-IR(cm^{-1}) 1637, 1585 (C=N), 1546 ($C{=}N_{triazin}$).

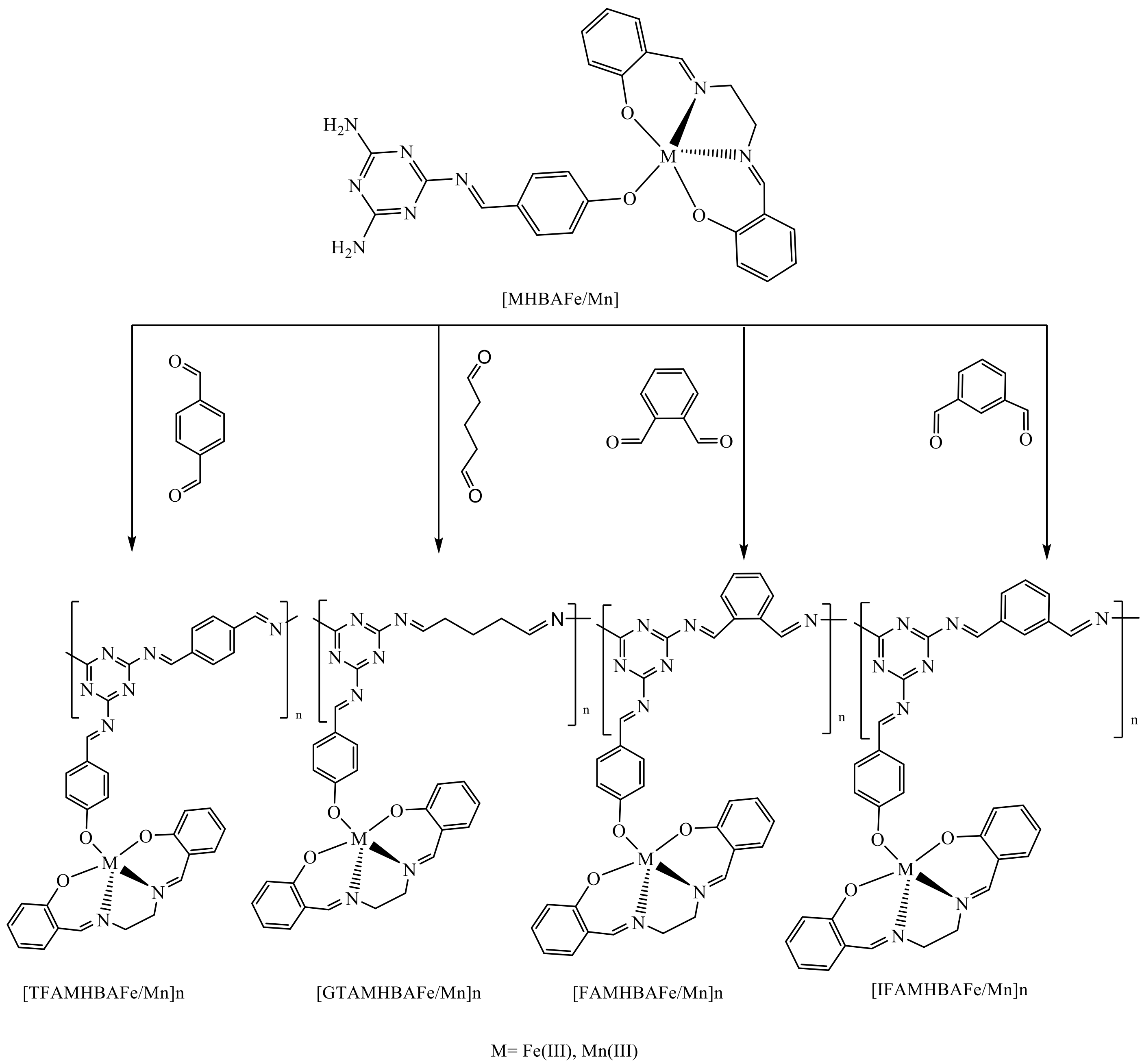

Figure 5. $[TFAMHBAFe/Mn]_n$, $[GTAMHBAFe/Mn]_n$, $[FAMHBAFe/Mn]_n$, $[IFAMHBAFe/Mn]_n$.

3. Results and Discussion

In this study, 2,4,6-triamino-1,3,5-triazine (Melamine) and 4-hydroxybenzaldehyde [HB] were used as starting material and heterocyclic Schiff base monomer 4-((4,6-diamino-1,3,5-triazine-2-imino)methyl)phenol [MHBA] was synthesized.

Single oxygen coordinated bridged monomer complex structures obtained with synthesized [MHBA] and [Fe(III)/Mn(III)(salen)Cl] complexes are terephthalaldehyde [TFA], glutaraldehyde [GTA], phthalaldehyde [FA] and isophthalaldehyde [IFA] Schiff base polymer complexes were obtained.

In the ^{1}H NMR spectrum of the [MHBA] monomer ligand OH protons, a corresponding singlet chemical shift value of 9.76 ppm was observed. In addition, it was observed that CH=N singlet chemical shift values occurred at doublet 7.74-7.72 and 6.89-6.91 ppm and 8.39 ppm. The singlet corresponding to the amine peaks was observed at 6.11 ppm (Figure 6) (Tahmassebi and Sasaki, 1998).

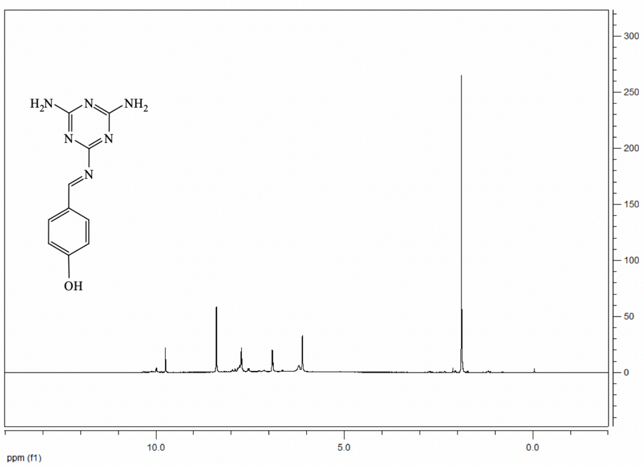

Figure 6. ^{1}H NMR spectrum of [MHBA].

FT-IR spectra of the compounds were taken. The FT-IR spectral data of the obtained monomer ligand and their Fe(III) and Mn(III) complexes are given in the experimental section. When we examine these values; OH peaks that are not present in the precursors of the monomer ligand were observed as a result of the condensation reaction with p-hydroxybenzaldehyde, as a result of the condensation reaction, the C=N group was 1649 cm^{-1} next to the new peak of 3324 cm^{-1} (Figure 7). In addition, it has been observed that the OH peaks of the bridged compounds coordinated with single oxygen with the [Fe(III)/Mn(III)(salen)Cl] complexes, which we have synthesized using the literature, have disappeared in the amine vibrations seen in the complexes of the monomer ligand at 3202 and 3172 cm^{-1}, respectively. It was also observed that M-O and M-N bonds in Salen complexes were at 780-810 cm^{-1} and 733-677 cm^{-1}, respectively (Figure 8). The resulting unidirectional Fe(III), Mn(III) complexes were polymerized with terephthalaldehyde, glutaraldehyde, phthalaldehyde and isophthalaldehyde in the presence of K_2CO_3 under reflux in ethanol. As a result of the condensation reaction, the C=N group was 1687, 1592-1675, 1587/1623, 1597-1623, 1596/1687, 1592-1675, 1587/1624, 1597-1637,1585 cm^{-1}, respectively (Figure 9-13) (Koc and Ucan, 2007).

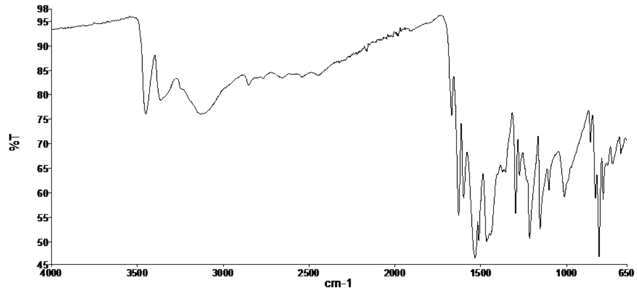

Figure 7. [MHBA]'s FT-IR spectrum.

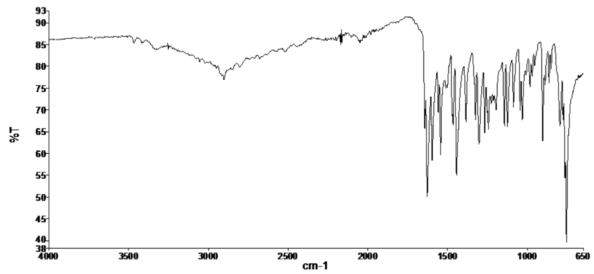

Figure 8. [MHBAFe(III)] FT-IR spectrum.

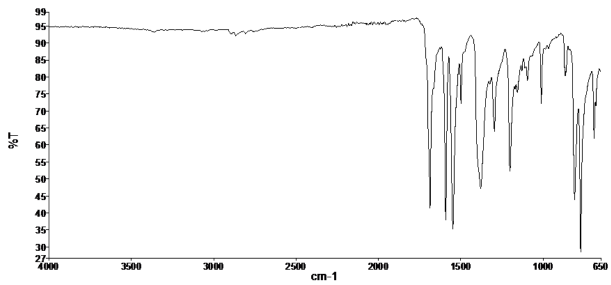

Figure 9. $[TFAMHBAFe(III)]_n$ FT-IR spectrum.

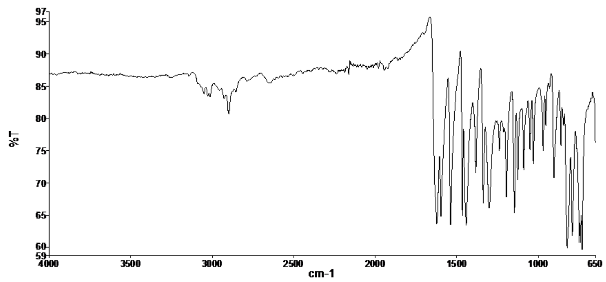

Figure 10. $[GTAMHBAFe(III)]_n$ FT-IR spectrum.

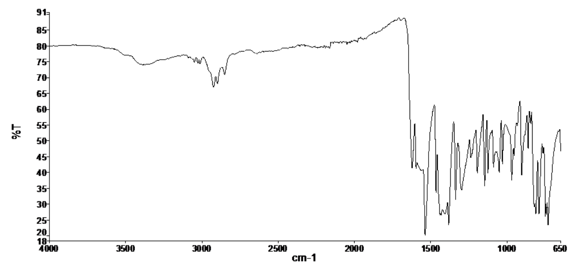

Figure 11. $[GTAMHBAMn(III)]_n$ FT-IR spectrum.

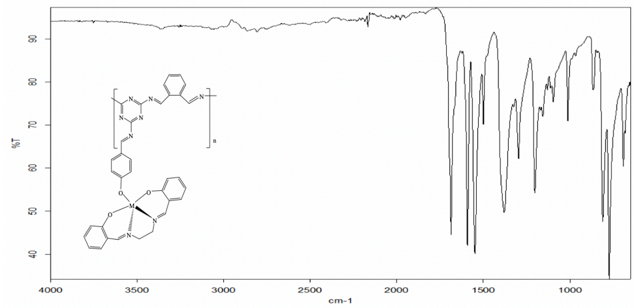

Figure 12. $[FAMHBAFe(III)]_n$ FT-IR spectrum.

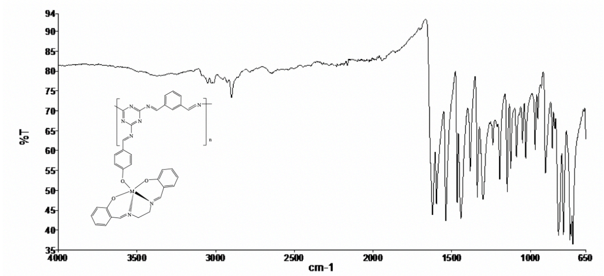

Figure 13. $[IFAMHBAFe(III)]_n$ FT-IR spectrum.

Synthesized [MHBA] and Fe(III), Mn(III) complexes [MHBAFe/Mn] were obtained with weak field effect for the BM values of 5.27 and 4.36, respectively, and $t_{2g}^{5}eg^{o}$, $t_{2g}^{3}eg^{1}$ were observed, respectively. As a result, it was estimated to have a triangular pyramidal (dsp^3) geometric structure, since it showed a weak field complex feature. As a result, since the complex structures have the d^5 and d^4 electron configurations calculated theoretically, it is estimated to have a triangular bipyramid geometry in sp^3d and dsp^3 hybridization, since they show weak ligand properties (Table 1) (Koc and Ucan, 2008).

Flow rates were determined using a 15 mL Oswald viscometer for the molecular weight of the synthesized [MHBA] and $[IFAMHBAFe/Mn]_n$ polymer compounds. The flow rates of [MHBA], the acetone solution used for the subsequent monomer (1% melamine) and polymer (1% $[IFAMHBAFe/Mn]_n$) in this solution were determined.

Table 1. Physical Properties of Monomer, Polymer and Salen Complexes

Compounds	Color	Yield (%)	M.P. (°C)	μ_{eff} (B.M.) 298 K	Found (Calculated) (%)				
					C	H	N	Fe	Mn
$C_{10}H_{10}N_6O$ MHBA	White	80	183	-	53.17 (52.17)	4.75 (4.38)	36.48 (36.50)	-	-
$C_{27}H_{26}FeN_8O_3$ [MHBAFe(III)]	Black	76	160	5.27	57.09 (57.26)	4.98 (4.63)	19.83 (19.72)	9.38 (9.86)	-
$C_{27}H_{26}MnN_8O_3$ [MHBAMn(III)]	Black	79	184	4.36	57.25 (57.35)	4.12 (4.63)	19.05 (19.82)	-	9.80 (9.72)
$C_{37}H_{34}FeN_8O_3$ $[TFAMHBAFe(III)]_n$	Brown	80	240	5.64	63.67 (63.98)	4.36 (4.93)	16.98 (16.13)	8.32 (8.04)	-
$C_{37}H_{34}MnN_8O_3$ $[TFAMHBAMn(III)]_n$	Brown	82	250	4.33	63.25 (64.07)	3.96 (4.94)	16.86 (16.15)	-	6.80 (7.92)
$C_{34}H_{36}FeN_8O_3$ $[GTAMHBAFe(III)]_n$	Yellow	78	267	5.68	60.65 (61.82)	4.63 (5.49)	15.36 (16.96)	9.43 (8.45)	-
$C_{34}H_{36}MnN_8O_3$ $[GTAMHBAMn(III)]_n$	Yellow	76	2340	4.35	60.18 (61.91)	4.02 (5.50)	17.33 (16.99)	-	7.56 (8.33)
$C_{37}H_{34}FeN_8O_3$ $[FAMHBAFe(III)]_n$	Yellow	64	294	5.29	62.65 (63.98)	3.72 (4.93)	15.97 (16.13)	9.65 (8.04)	-
$C_{37}H_{34}MnN_8O_3$ $[FAMHBAMn(III)]_n$	Yellow	67	260	4.31	63.04 (64.07)	5.87 (4.94)	17.34 (16.15)	-	8.90 (7.92)
$C_{37}H_{34}FeN_8O_3$ $[IFAMHBAFe(III)]_n$	Orange	76	235	526	62.87 (63.98)	5.45 (4.93)	15.02 (16.13)	9.12 (8.04)	-
$C_{37}H_{34}MnN_8O_3$ $[IFAMHBAMn(III)]_n$	Orange	75	278	4.37	65.02 (64.07)	5.67 (4.94)	17.09 (16.15)	-	8.88 (7.92)

In light of the results obtained, a lower flow rate than expected occurred as a result of breaking these interactions with the addition of monomer, which reduces the flow rate of hydrogen bonds between HCl and H_2O molecules (Table 2) (Arslan, 2015).

Table 2. Viscometer

Compounds	Flow Rate
Aceton	0.36 s
Monomer Ligand	0.35 s
Polymer Ligand Complex	36.54 s

TGA measurement of monomeric Schiff base complexes obtained with synthesized [MHBA], Fe(III) ligand complex [MHBAFe] between 0-900 °C, in 20 °C/min^{-1} N_2 atmosphere was made. According to the TGA diagram of the monomeric Schiff base Fe(III) ligand complex [MHBAFe], gaseous H_2O, CO_2, C_6H_6, N_2 and H_2 are first removed from the environment and at 155, 325 and 455 °C, 64.32% (Theoretical: 65.46%) three-step. It is observed that the decomposition reaction that takes place is a total mass loss. However, at 800-880 °C, the mass loss of matter continues. It is estimated that this is due to the presence of the triazine ring and metal oxides in the environment (Figure 14) (Karipcin and Karatas, 2001).

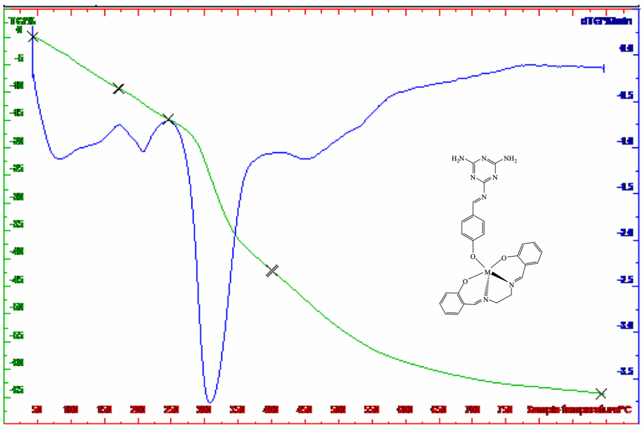

Figure 14. [MHBAFe(III)] TGA spectrum.

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CRediT author statement

Nihal YILDIRIM: Yazılım, Görselleştirme, Kaynaklar, Yazma-İnceleme, Düzenleme

Ziya Erdem KOÇ: Kavramsallaştırma, Metodoloji, Veri iyileştirme, Yazma-Özgün taslak hazırlama, Denetleme, Yazma-İnceleme, Düzenleme.

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