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The Synthesis of Microwave Assisted Melamine-Schiff Bases and Investigation of Bridged Fe(III) Metal Complexes

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Abstract: In this study, 2,4,6-triamino-s-triazine (melamine) as the starting material. Microwave assisted Schiff base reaction took place with the condensation reaction of melamine and 3-hydroxybenzaldehyde. Then, s-triazine-core Schiff base ligand complexes were obtained by obtaining a single oxygen-bridged compound of [(Fe(Salophen)Cl] ligand complexes, which we synthesized Schiff base s-triazine by literature. The obtained multidirectional s-triazine-core Schiff base ligand complexes were under microwave assisted with 3-hydroxybenzaldehyde compounds. The condensations proceed in short time to give products which, in certain instances, are not readily attainable by conventional condensation techniques. Due to its good performance, the microwave device used in household was preferred. Consequently, the structures of the obtained ligand and complexes were characterized using elemental analysis, FT-IR spectroscopy, ^{1}H-NMR spectroscopy and magnetic susceptibility measurement techniques.

Keywords: Melamine, Schiff base, s-Triazine, Salophen, Microwave

Introduction

Melamine, which is one of its compounds, is rapidly increasing in coordination chemistry, environmental chemistry, biochemistry, polymer chemistry, pharmaceutical chemistry and electronics industry (Nozha et al., 2021; Uysal et al., 2012). In addition, Melamine Schiff base compounds are used in medicine, especially as molecular magnetic material, and such heterocyclic compounds are used as active ingredients of antitumor and anticancer drugs (Arslaner et al., 2017; Koc & Uysal, 2016; Portalone, 2008). Melamine compounds have gained importance in environmental chemistry in the storage of gases with metal-organic lattice structures (Qian & Huang, 2010; Yu et al., 2008).

Schiff bases are very important structures condensation products of primary amines with aldehydes for synthetic organic chemistry (Dhar & Taploo, 1982; Schiff, 1869). Schiff-base is considered a dynamic compound with a reversible imine bond derived from the dehydration reaction between amine and carbonyl groups (Kopel, Sindelar, Biler, et al., 1998; Zabardasti & Shangaie, 2016; Zhang et al., 2021). Over the years, Schiff bases and metal complexes have played an important role in the development of coordination chemistry, chelating ligands and complex biochemical reactions as they readily form stable complexes with most of the transition metals (Jayabalakrishnan et al., 2002; Kocyigit et al., 2010; Sindhu et al., 2013). In recent years, the widespread use of Schiff bases and metal complexes, especially in various qualitative and quantitative analyses, enrichment of radioactive materials, pharmacological properties, the paint industry, and plastics industry has further increased, and it has attracted much attention due to their biochemical activities (Koc, 2011; Koc & Ucan, 2007; Koc & Uysal, 2010, 2011; Li et al., 2021; Naz et al., 2013; Uysal & Koc, 2010).

Nitrogen-containing heterocycles are highly important due to their classification as a significant group of synthetic and natural compounds(Thamer Abd Rehan, 2024). Then, the obtained microwave assisted s-triazine monomer was coordinated with the salophen ligand complex with a single oxygen and monopodal melamine centered monomer complexes were obtained (Celikbilek & Koc, 2014; Hao et al., 2021). Microwave-assisted

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organic synthesis (MAOS) continues to affect synthetic chemistry significantly by enabling rapid, reproducible experiments. We envisioned that microwave irradiation would enhance this chemistry and expand the chemistry scope (Celik et al., 2019; Diaz-Ortiz et al., 2001).

Experimental

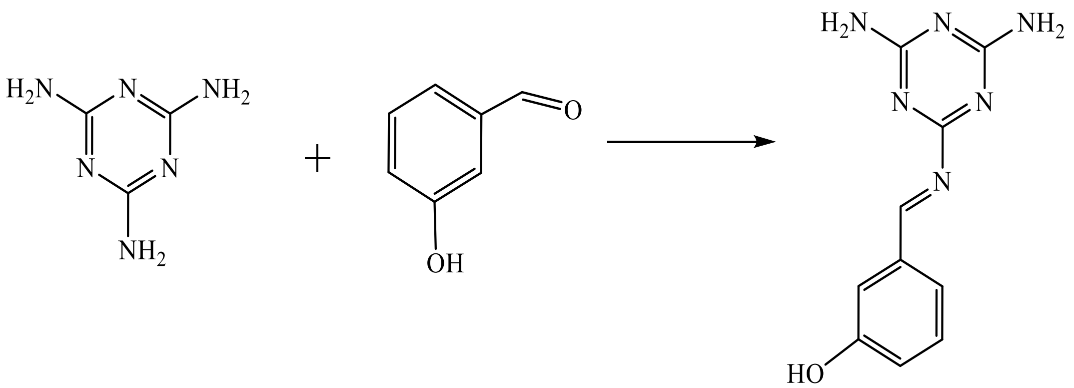
Materials and Methods

The chemicals were purchased from Aldrich and Merck was used as received. LG-health wave microwave system (MG-607APR, 230V—50Hz) was used and the output of microwave power is mentioned as percent intensity i.e. (20%, 40%, 60%, 100%). 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 analyser (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, B.M., where χM is the molar susceptibility (Cambridge, UK). TGA analyses of the compounds were performed on the Mettler Tole *Third Level Headings*

Preparation of Microwave Assisted 3-((4,6-Diamino-1,3,5-triazine-2-imino)methyl)Phenol (DTMP)

Melamine was suspended in 60 mL of benzene (1 mmol, 1.26 g). Then 3-hydroxybenzaldehyde (1 mmol, 1.22 g) was added to the mixture piece by piece. The mixture was heated using microwave irradiation technique at 350W for 25 minutes, permitted to cool, and poured in a thin stream into a large volume of rapidly stirred water. To separate the precipitated monomer, the mixture was cooled and the solid was filtered apart. The resulting white precipitate was dried. **$C_{10}H_{10}N_6O$:** ^{1}H NMR (DMSO-d_6, ppm): 10.64 (s, H, OH), 9.71 (s, H, CH=N), 8.55 (s, H, Ar-H), 7.83-6.88 (m, 3H, Ar-H). FT-IR (cm^{-1}) 3467-3414 (NH_2), 3167 (OH), 1649 (C=N), 1526 ($C=N_{triazine}$).



3-((4,6-diamino-1,3,5-triazin-2-imino)methyl)phenol

Figure 1. Proposed structures of the microwave-assisted (DTMP) ligand

Synthesis of Salophen Ligand and Salophen Complexes

The synthesis of Salophen ligand and Salophen complexes has been synthesized according to the mentioned literature. (Gembicky et al., 2000; Kopel, Sindelar, & Klicka, 1998).

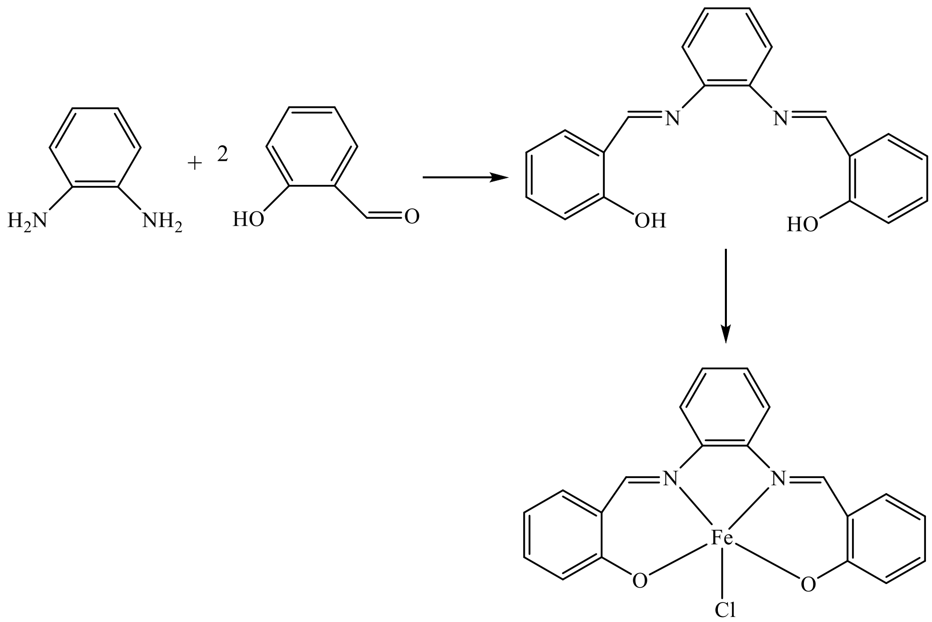


Figure 2. Salophen and [Fe(salophen)Cl] complex

3-((4,6-diamino-1,3,5-triazine-2-imino) Methyl)phenol [Fe(salophen)Cl] Complex

Suspension solution was prepared in the resulting monomer (1 mmol, 0.23 g) ethanol. [Fe(salophen)Cl] (1 mmol, 0.37 g) dissolved in ethanol was added to the complex compound monomer. The reaction mixture was stirred for four hours at 100 °C under a back cooler. The reaction solution was filtered and dried in a sedimentary oven. FT-IR (cm^{-1}) 3317-3354 (NH_2), 1635 (C=N), 1546 ($C=N_{triazin}$).

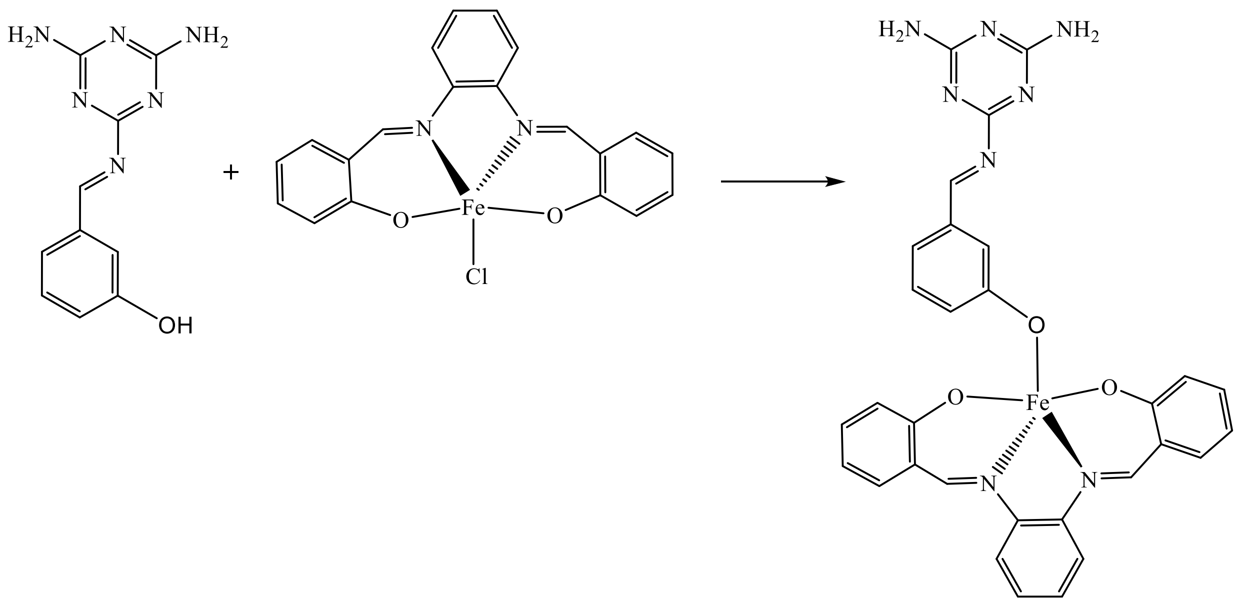


Figure 3. Monopodal Schiff base ligand [Fe(salophen)(DTMP)] complex

Tripodal Schiff Base Ligand [Fe(salophen)(DTMPDHB] Complex

[Fe(salophen)(DTMP)] (1 mmol, 0.85 g,) was dissolved in 30 mL of methanol and stirred under reflux for 2 h. 2,5-Dihydroxybenzaldehyde (2 mmol, 0.28 g) 20 mL methanol was added to the resulting mixture. The mixture was reflux at 4 h and 5 drops of acetic acid catalyst were added. It was mixed for a while until the powder formed, and a color change was observed. The precipitate was filtered. [SALMHBAFe(III)(salophen)]: FT-IR (cm^{-1}) 3240, 3175 (OH), 1642,1624 (C=N), 1553 ($C=N_{triazine}$).

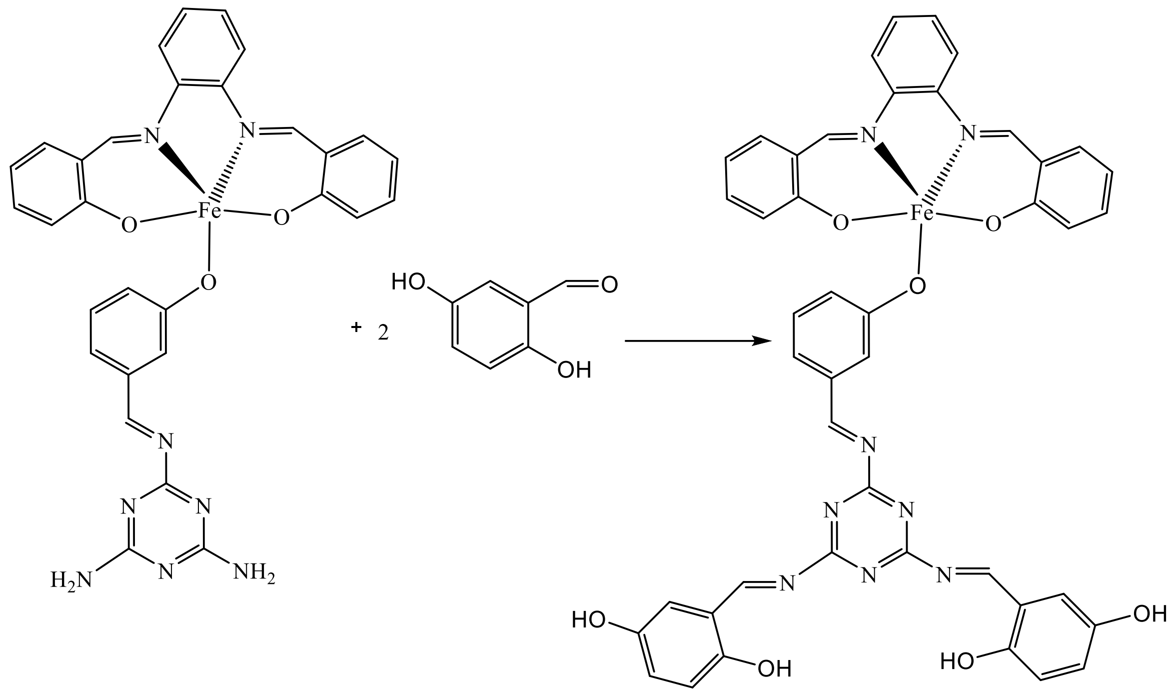


Figure 4. Tripodal Schiff base ligand [Fe(salophen)(DTMPDHB] complex

Tripodal Schiff Base Ligand [Fe_2(salophen)(DTMPDHB)] Complex

Suspension of [Fe(salophen)(DTMPDHB] ligand complexes (1 mmol 0.85 g) in 20 mL of ethanol was prepared in a 100 mL flask on $FeCl_3$ (1 mmol 0.16 g) were added in 20 mL ethanol. boil under a back cooler for 3 h at around 80 ºC. The solvent was evaporated by half and allowed to cool (under room conditions). Then, half of the water was added, left for a day, filtered in a vacuum, washed with water and dried in an oven at 105 ºC. [Fe_2(salophen)(DTMPDHB)]: FT-IR (cm^{-1}) 1666,1634, (C=N), 1543.

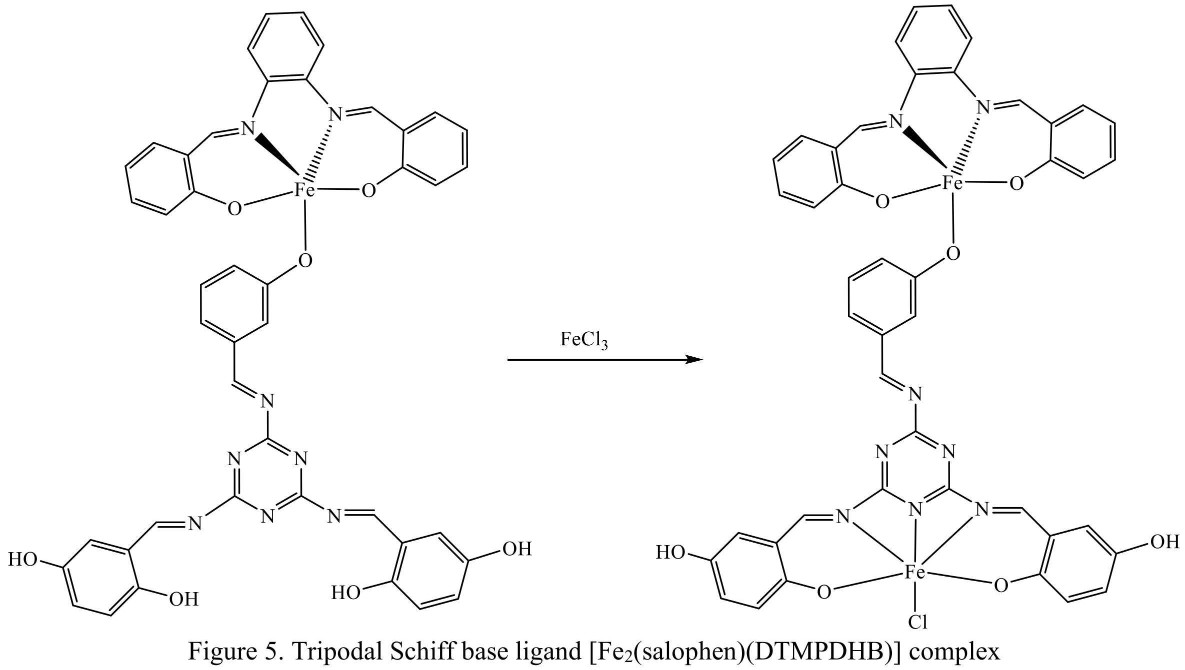
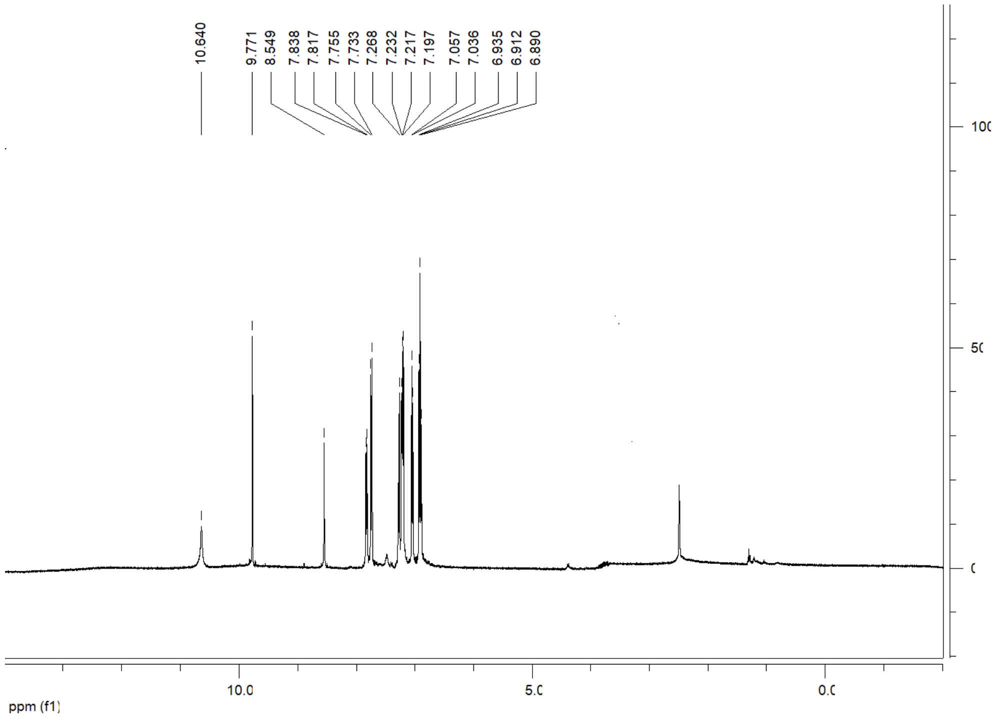


Figure 5. Tripodal Schiff base ligand [Fe_2(salophen)(DTMPDHB)] complex

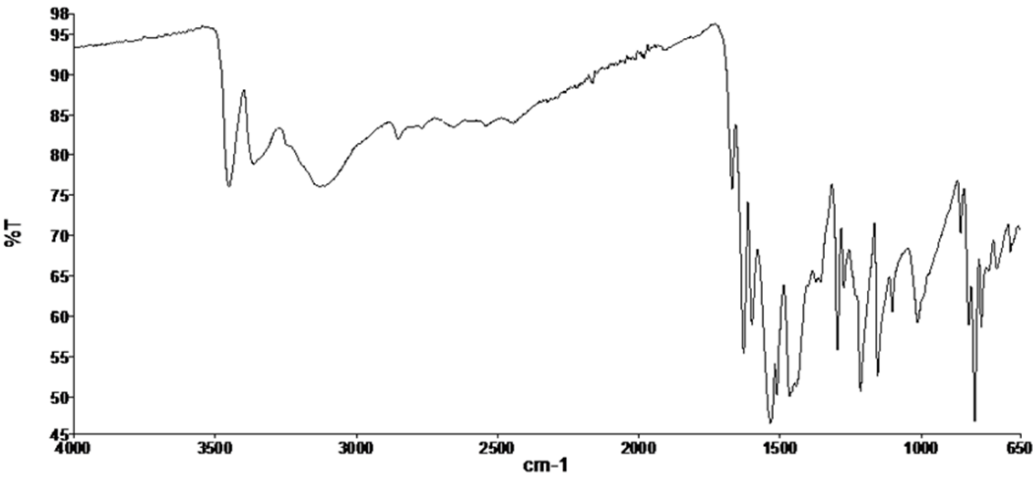
Results and Discussion

In this study, melamine Schiff base monomer 3-((4,6-diamino-1,3,5-triazine-2-imino)methyl)phenol (DTMP) was synthesized by the reaction of Melamine and 3-hydroxybenzaldehyde [HB], which was used as the output material. A single oxygen-coordinated bridged monomer complex obtained with the synthesized (DTMP) and [Fe(salophen)Cl] complex was obtained [Fe(salophen)(DTMP)] complexes are 2,5-dihydroxybenzaldehyde Schiff base melamine complexes [Fe_2(salophen)(DTMPDHB)] were obtained. ^{1}H NMR spectrum of the monomer ligand (DTMP) observed a corresponding OH protons in the singlet chemical shift value of 10.64 ppm. In addition, aromatic CH was observed to be 8.55/7.83-6.88 ppm and 9.71 ppm respectively at CH=N singlet chemical shift.(Tahmassebi & Sasaki, 1998).



Scheme 1. ^{1}H NMR spectrum of the monomer ligand (DTMP)

FT-IR spectra of the compounds were obtained. The obtained monomer ligand and their FT-IR spectral data of the Fe(III) complex are given in the experimental section. When we examine these values, OH peaks, which were not found in the input materials of the monomer ligand, were observed as OH 3167 cm^{-1} because of the condensation reaction with 3-hydroxybenzaldehyde, as well as a new peak of OH 3167 cm^{-1} in the C = N group 1649 cm^{-1}. In addition, amine vibrations seen in the complexes of the monomer ligand at 3467 and 3414 cm^{-1} were synthesized using the literature and it was observed that the OH peaks of the bridged compounds coordinated with a single oxygen were lost. In Salophen complexes, M-O and M-N bonds were also observed at 760-830 cm^{-1} and 753-685cm^{-1}, respectively (Koc & Ucan, 2007).



Scheme 2. FTIR spectrum of the monomer ligand (DTMP)

The synthesized (DTMP) and Fe(III) complex [Fe(salophen)(DTMP)], [Fe(salophen)(DTMPDHB] and [Fe_2(salophen)(DTMPDHB)] obtained compounds were observed in $t_{2g}^3e_g^2$, with weak field effects for BM values of 5.27/5.30/5.05, respectively. As a result, it was estimated that it had a triangular pyramidal (dsp^3) geometric structure because it showed a weak field complex feature. As a result, since the complex structure has a theoretically calculated d^5 electron configuration and shows weak ligand properties, it is estimated that it has a triangular bipyramid geometry in sp^3d hybridization (Table 1.) (Koc & Ucan, 2008).

Table 1. Physical properties of monopodal, tripodal and metal complexes

Compounds	Color	Yield (%)	M.P. (°C)	μ_{eff}	Found (Calculated) (%)		
					C	**H**	**N**
(DTMP)	White	80	183	-	53.17 (52.17)	4.75 (4.38)	36.48 (36.50)
[Fe(salophen)(DTMP)]	Black	75	160	5.27	60.49 (60.60)	4.38 (4.27)	18.33 (18.24)
[Fe(salophen)(DTMPDHB]	Orange	65	300*	5.30	63.75 (63.24)	4.05 (4.01)	13.65 (13.11)
[Fe_2(salophen)(DTMPDHB)]	Yellow	60	300*	5.05	57.47 (57.26)	3.66 (3.42)	11.78 (11.87)

* Decomposition

Conclusion

In this study, Melamine was used as the starting material. Melamine reacted with the addition of 2,5-dihydroxybenzaldehyde in the presence of 1,4-dioxane to obtain 3-((4,6-Diamino-1,3,5-triazine-2-imino)methyl) phenol (DTMP) complexation was carried out with the obtained [Fe(salophen)(DTMP)], anhydrous $FeCl_3$. In addition, Melamine reacts with 2,5-dihidroxybenzaldehyde was [Fe(salophen)(DTMPDHB] obtained. Schiff base compounds with different donor groups were synthesized by the condensation of these aldehyde and amine group molecules. Its bidirectional [Fe_2(salophen)(DTMPDHB)]by hydroxyl groups.

Recommendations

The introductory substances used in this study are ligands and complexes that are newly studied and not encountered in literature. We think that the obtained ligands and complexes will gain importance especially in terms of environmental chemistry. In addition, in our studies that progress as molecular magnetic materials due to their magnetic properties, new and more usable properties will be examined by focusing more on this issue.

Scientific Ethics Declaration

*The author declares that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the author.

Conflict of Interest

*The author declares that there is no conflict of interest related to this work.

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