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Synthesis and Characterization of S-Triazine-Schiff Bases and Transition Metal Complexes

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Abstract: In this study, a new synthetic route for preparing multidirectional ligands was developed by using as starting material 2-chloro-4,6-diamino-1,3,5-triazine (CDAT) core. In the synthesis of new triazine centered dipodal Schiff bases have been synthesized via condensation reactions between 1 equiv. (CDAT) and 2 equiv. aldehyde molecules according to the methods mentioned in published literature. (CDAT) has been reacted with 2-hydroxybenzaldehyde (SA) molecule in the presence of acetic acid and acetone to get 2-chloro-4,6-N,N'-bis(2-hydroxybenzylidene)-1,3,5-triazine (CDATSA). On the other hand, pentadentate (ONNNO) Schiff bases, mononuclear $[\text{Fe}(\text{CDATSA})\text{Cl}]$ have been gained via the reaction with nonaqueous FeCl_3 . The complexes were characterized as high spin d5 ($S = 5/2$) distorted trigonal bipyramidal. As a result, ligand has been characterized by FT-IR and ^1H -NMR. Structures of isolated monomers have been clarified with the analysis of FT-IR and elemental analysis and data of magnetic susceptibility.

Keywords: Schiff base, Dipodal, 2-Chloro-4,6-diamino-1,3,5-triazine, Metal complexes.

Introduction

Schiff bases, also referred to as imines, constitute an important class of compounds formed through the condensation of primary amines with aldehydes or ketones, making them valuable intermediates in synthetic organic chemistry (Koç, 2011). The characteristic imine ($\text{C}=\text{N}$) linkage is generated via a reversible dehydration reaction between amino and carbonyl groups, imparting dynamic covalent behavior to these molecules. Owing to their excellent coordination ability, Schiff bases readily chelate a wide variety of transition metal ions, and their corresponding metal complexes have become indispensable in coordination chemistry and bioinorganic research. Extensive investigations over recent decades have demonstrated that Schiff bases and their metal complexes possess broad practical importance (Jayabalakrishnan et al., 2002). They have found applications in analytical chemistry, radioactive material separation, medicinal chemistry, polymer and dye industries, and plastic manufacturing. In addition, their diverse biological and pharmacological properties have attracted considerable scientific interest (Arslaner et al., 2017; Ugras et al., 2006). Iron(III) coordination compounds have remained an active research area over the past two decades, particularly because high-spin Fe(III) complexes exhibit interesting magnetic and structural properties (Gembicky et al., 2000). Our previous studies have focused on the synthesis and characterization of several s-triazine-based coordination compounds (Koç & Uysal, 2010; Uysal & Koç, 2010). Building upon these investigations, the present work describes the preparation of novel Fe(III) complexes incorporating Schiff base ligands derived from CDAT. The Schiff base ligand (CDATSA) was synthesized through the condensation reaction between CDAT and the corresponding salicylaldehyde (SA), as illustrated in Scheme 1. Subsequent coordination of CDATSA with FeCl_3 afforded the mononuclear high-spin Fe(III) complex, $[\text{Fe}(\text{CDATSA})\text{Cl}]$ (Scheme 2). The synthesized ligands and their corresponding complexes were comprehensively characterized using elemental analysis, FT-IR spectroscopy, ^1H NMR spectroscopy, and magnetic susceptibility measurements, confirming the proposed molecular structures.

Experimental

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). ^1H 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 $\text{Hg}[\text{Co}(\text{SCN})_4]$ as a calibration by the constant magnetic field. The effective magnetic moments, μ_{eff} , per metal atom were calculated from the expression, $B.M.$, where χ_M is the molar susceptibility (Cambridge, UK). TGA analyses of the compounds were performed on the Mettler Toled *Third Level Headings*

Synthesis of 2-Chloro-4,6-*N,N'*-bis(2-hydroxybenzylidene)-1,3,5-triazine (DACTSA)

2-Hydroxybenzaldehyde (2 mmol, 0.27 g) is dissolved in 30 mL of acetone. 2 drops of acetic acid are added and mixed. Then, 2-chloro-4,6-diamino-1,3,5-triazine (1 mmol, 0.14 g) is dissolved in 15 mL of acetone and added to the solution. The mixture is stirred at 90°C for 8 hours. When the reaction is complete, the light green precipitate is separated with filter paper, leaving behind a blue band. The obtained filtrate is left to crystallize. After filtration, light green powder is obtained and dried in an oven. M.P.: 130 °C; 87%, FT-IR (cm^{-1}) 3492 (OH), 1655 (CH=N), 1578 (C=N_{triazin}). ^1H NMR ($\text{DMSO}-d_6$) δ 10.88 (s, 2H), 10.61 (s, 2H), 9.82 (s, 2H), 7.51-6.30 (m, 6H).

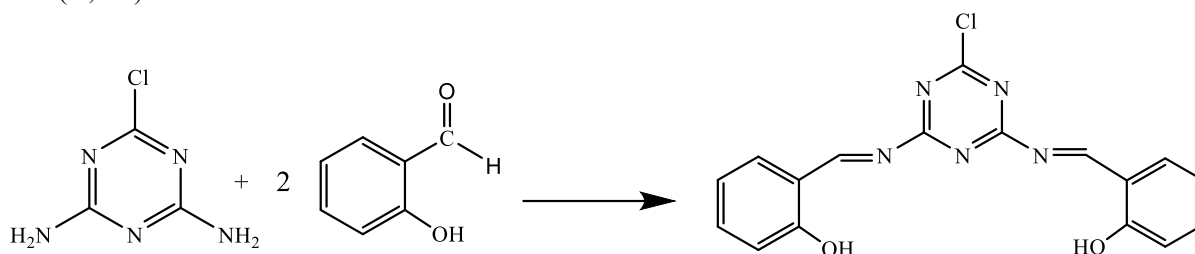


Figure 1. Synthesis of (CDAT) ligand

The Fe(III) complex of 2-chloro-4,6-*N,N'*-bis(2-hydroxybenzylidene)-1,3,5-triazine [$\text{Fe}(\text{CDATSA})\text{Cl}$]

(CDATSA) (1 mmol, 0.38 g) is dissolved in 20 mL of acetone. Then, FeCl_3 (1 mmol, 0.16 g) solution dissolved in 20 mL ethanol is added. It is mixed under reflux at 100 °C for 4 h. The resulting black precipitate is filtered and dried in a vacuum desiccator. E.n.: >350 °C; %70, FT-IR (cm^{-1}) 1650 (CH=N), 1570 (C=N triazin).

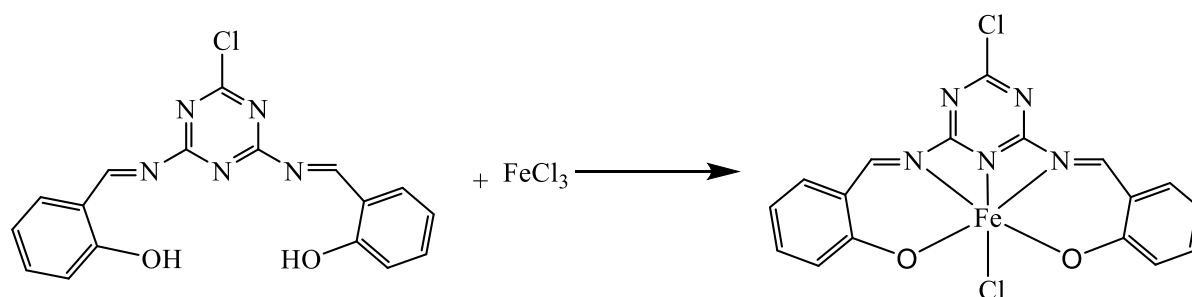
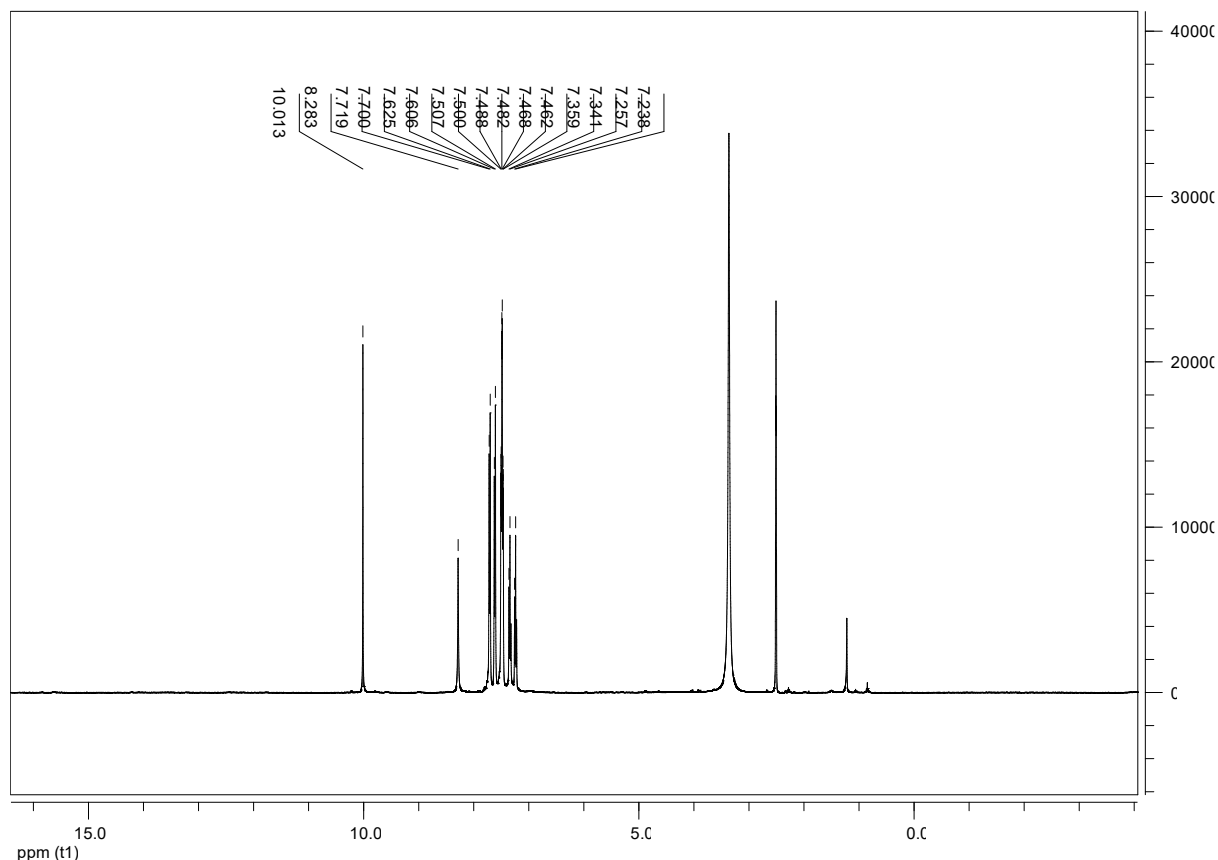


Figure 2. [$\text{Fe}(\text{CDATSA})\text{Cl}$] complex

Results and Discussion

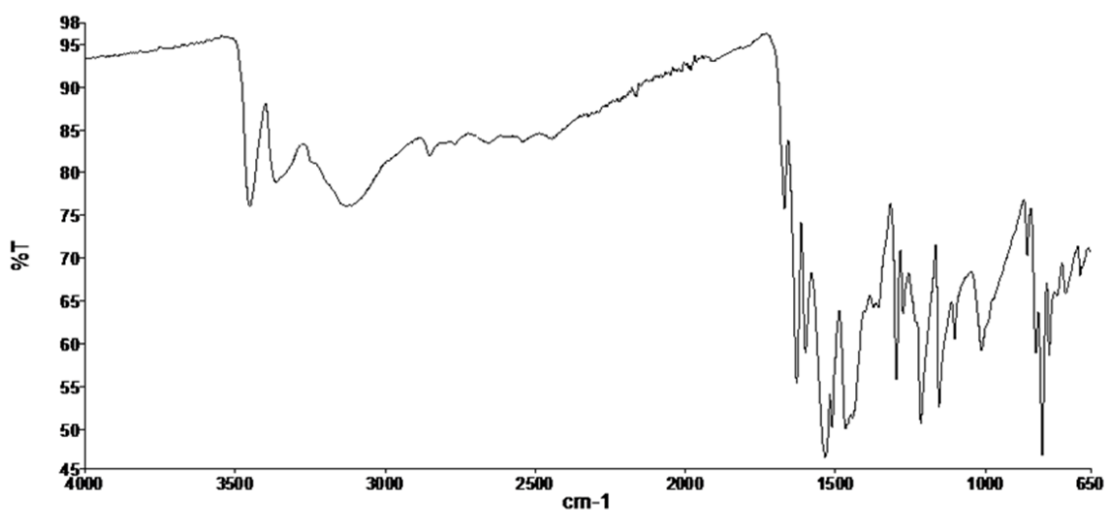
In this study, heterocyclic Schiff base monomer 2-chloro-4,6-*N,N'*-bis(2-hydroxybenzylidene)-1,3,5-triazine (CDATSA) was synthesized by the reaction of 2-chloro-4,6-diamino-1,3,5-triazine (CDAT) and 2-hydroxybenzaldehyde [SA], which was used as the output material. A single oxygen-coordinated monomer

complex obtained with the synthesized $[\text{Fe}(\text{CDATSA})\text{Cl}]$ complex was obtained. ^1H NMR spectrum of the monomer ligand OH protons (DACTSA) observed a corresponding singlet chemical shift value of 10.01 ppm. In addition, aromatic CH was observed to be 7.24-7.72 ppm and 8.28 ppm respectively at $\text{CH}=\text{N}$ singlet chemical shift. (Tahmassebi & Sasaki, 1998).



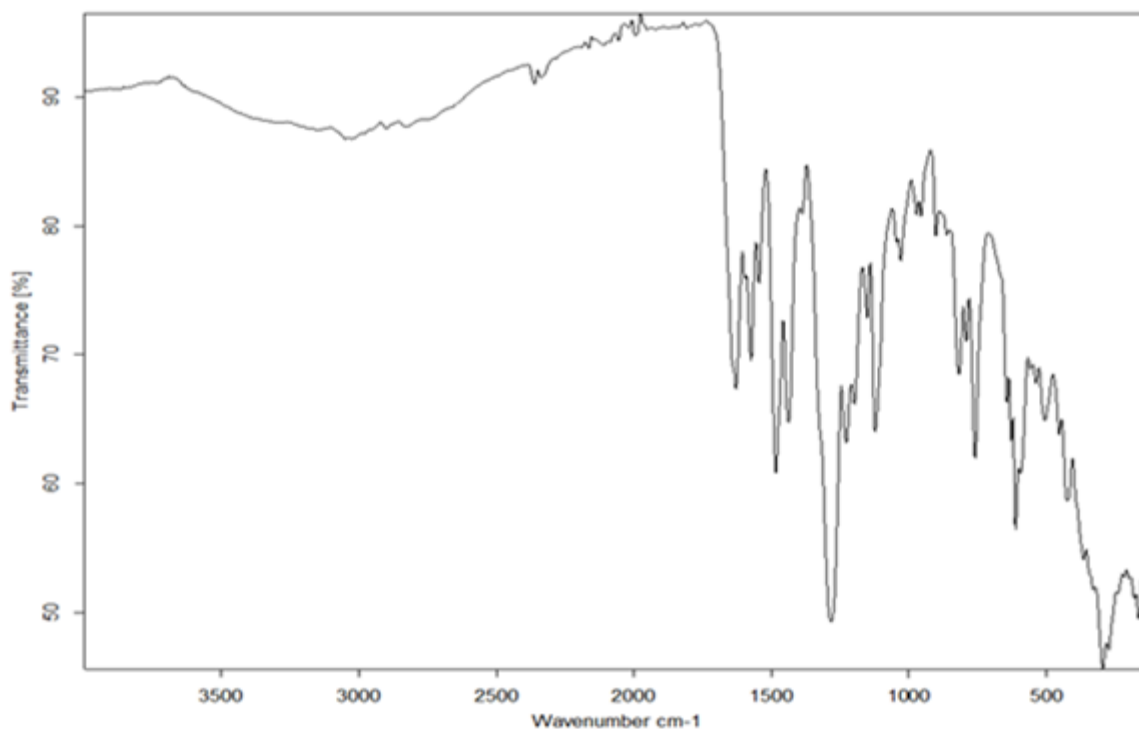
Scheme 1 ^1H NMR spectrum of the monomer ligand (CDAT)

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 $\text{OH } 3492 \text{ cm}^{-1}$ because of the condensation reaction with 2-hydroxybenzaldehyde in the $\text{C}=\text{N}$ group 1655 cm^{-1} . In addition, it was observed that the OH peaks of the compounds coordinated with a single oxygen were lost. In Salen complexes, M-O and M-N bonds were also observed at $750\text{-}840 \text{ cm}^{-1}$ and $755\text{-}680 \text{ cm}^{-1}$, respectively (Koc ve Ucan, 2007).



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

All the magnetic moments of the complexes were measured at room temperature. Based on spectral evidence, the magnetic moment values metal atom of monomer $[\text{Fe}(\text{CDATSA})\text{Cl}]$ complex show paramagnetic behavior with a magnetic susceptibility value atom: 5.25, respectively. B.M. in which the Fe(III) cations have an approximately octahedral environment. The magnetic behavior of Fe(III) complexes is in good harmony with the proposed mononuclear structures. (Koc & Ucan, 2008).



Scheme 3. FTIR spectrum of the $[\text{Fe}(\text{CDATSA})\text{Cl}]$

Conclusion

In this study, 2-chloro-4,6-diamino-1,3,5-triazine (**CDAT**) was used as the starting material. (**CDAT**) reacted with the addition of 2-hydroxybenzaldehyde in the presence of acetone to obtain 2-Chloro-4,6-N,N'-bis(2-hydroxybenzylidene)-1,3,5-triazine (**CDATSA**). Complexation was carried out with the obtained (**CDATSA**). anhydrous FeCl_3 . In addition, Schiff base compounds with different donor groups were synthesized by the condensation of these aldehyde and amine group molecules. Its monodirectional Fe(III) mono homonuclear complexes.

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.

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