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SCHIFF BASES AND THEIR POLYMER RESINS IN AQUEOUS SOLUTIONS: AN ANALYTICAL AND ADSORPTION EFFICIENCY STUDY

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Abstract: The goal of this study is to synthesize, characterize, and analyse a number of chelating polymer resins containing Schiff base ligands that can be utilized to remediate harmful metal-laden industrial wastewater. We create multiple polymer resins to find the ideal conditions for eliminating numerous metal ions. To avoid water pollution, industrial effluent must be cleaned before disposal because it includes high levels of hazardous metals. Several studies on various physical methods for separating, removing, and managing metal toxicants have been conducted. The condensation of 2-hydroxybenzaldehyde L1 and 1,3-benzodioxole-5-carboxaldehyde L2 resulted in the production of isoniazid Schiff base derivatives, specifically isonicotinic acid hydrazide. The azo-methane ligands were detected through the application of spectroscopic techniques, including infrared spectroscopy, mass spectrometry, and ^{1}H-NMR spectroscopy. Two different ways were used to make polymer resins: formaldehyde was used for L1 and methylene diphenyl diisocyanate (MDI) was used for L2. Thermal analysis was used to test the thermal stability of polymers. The cyclic voltammetry method was also used to investigate the electrochemical properties of the Schiff base ligands. Using time and pH, the chelating efficiency and loading capacity of generated ligands toward various metal ions were examined.

Keywords: Schiff bases, Metal ion adsorption, Polymer resins, Cyclic voltammetry.

Introduction

In recent years, using too much technological equipment's which led to severe produces damages into the environment. Metal ions are produced

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by a range of industrial operations, including mining, farming, battery production, and petroleum refining.[1,2] Heavy metals are naturally present in the environment, but human activities such as mining, industrialization, smelting, pollution, fertilizers, old water pipes, paints, and batteries have increased their abundance. Many of these metals are essential for living a healthy life. The main problem with these heavy metals is that they are not biodegradable, which means they can accumulate in live organs and creates serious health problems.[3]

Humans and environment are frequently affected when heavy metal ions are reach to higher levels than permitted for example, have been associated to respiratory, cardiovascular, hepatic, renal, and cancer illnesses.[4] This necessitates determining the populations of these species. Atomic absorption spectroscopy devices have various advantages over conventional analytical techniques for measuring metal ions.[5] Because metal ions have a predisposition to accumulate in living things and cause a variety of health problems even at low concentrations, industrial metal contamination of the environment is seen as a severe issue.[6] Many methods for removing metal ions from waste water have been proposed, including ion exchange, complexation, adsorption, and reverse osmosis.[7] Because of their significant selectivity for certain heavy metal ions, adsorption capabilities, reusability, sensitivity, and efficiency, chelating organic compounds have been the focus of numerous investigations on metal ion removal.[8,9]

Numerous studies have been conducted in an effort to develop chelating ligands that incorporate active groups, including imidazole, allyl thiourea, imine, and azo groups.[10-15] Furthermore, functionalization procedures have been used to alter chemicals and create novel chelating compounds. Schiff bases have been shown in studies to be important plant growth regulators, antimitotic agents,[16] antibacterial agents,[17] antioxidants, and anticorrosion agents.[18] Schiff bases are among the most commonly utilized organic ligands in coordination chemistry. Schiff bases can create coordination bonds with metal ions by employing an azo-methane group. They are often used in metal complexes because they are simple to synthesize. Schiff bases are frequently employed in biological activity, analytical chemistry, fungicidal action, the food and dye industries, and catalysis.

The fact that these compounds have enabled the employment of simple detection techniques for a wide range of organic and inorganic materials has increased interest in them as analytical chemistry reagents. Chelating polymers, often known as chelating sorbents, are polymer matrix probes with reactive donor groups. Dynamic clusters can synchronize with nearby ions. These polymers' sensitivity and selectivity depend on ligand type and media pH. Chelating polymers are used in heat-resistant fibres, protective coatings, ionic selective electrode membranes, environmentally friendly analytical reagents, and catalysts for oxidation, hydrogenation, hydrolysis, and polymeric initiators.[19]

Binding polymers have been extensively employed for the purpose of extracting heavy metal ions from environmental systems.[20] Improving active chelating polymers is critical for inorganic catalyst and metal ion recovery. These chelating polymers were found to exhibit high selectivity when compared to more traditional ion exchanger forms. These polymers have good physical and chemical properties, such as being strong, permeable, and having a large surface area.[21,22] The chemical structure of ligands and polymers containing recognition groups, such as the azo-methane group, is critical in improving chelating agent properties. When it comes to building compounds with heavy metal ions, Schiff bases are extremely sensitive and selective.[23,24]

A chelating agent from the reaction between isoniazid (isonicotinic acid hydrazide) and 2-hydroxybenzaldehyde (L1) and 1,3-benzodioxole-5-carboxaldehyde (L2) was used to synthesize new Schiff bases. These items detected copper, cadmium, and nickel ions. Cyclic voltammetry was used to study Schiff bases' electrochemical properties.

Experimental

Materials

Sigma Aldrich provided 2-hydroxybenzaldehyde, 1,3-benzodioxole-5-carboxaldehyde, and isonicotinic acid hydrazide acid. The components (solvents and chemicals) were used in their entirety, with no further purification.

Instrumentation

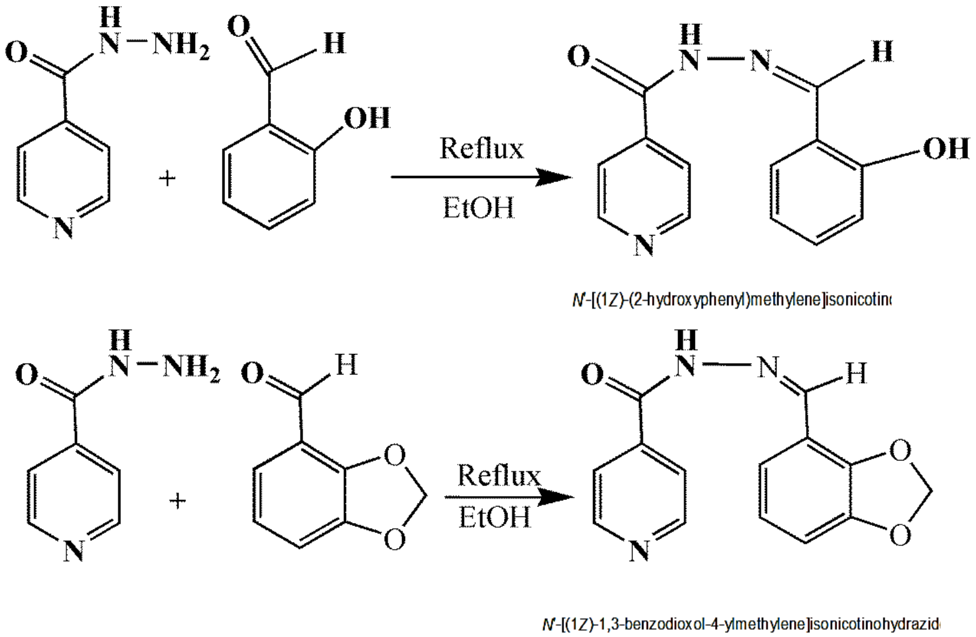
To record ^{1}H-NMR spectra, dimethyl sulfoxide (DMSO-d6) and (TMS) were utilized as standards. The Innova-500 Varian Device was used to collect ^{1}H-NMR readings. The EI (Electron impact) mass spectrum was measured using an Agilent Technologies-5975C device. The (DY2311-Potentiostat) gadget was used to record cyclic voltammetry measurements.

Synthesis of 2-hydroxyphenyl methylene isonicotionohydrazide, compound L_1[25]

Using a mixture of 50 mL of pure ethanol and 3 drops of concentrated acetic acid, 10 mmol of 2-hydroxybenzaldehyde (1.22 mL) was mixed with 10 mmol of isonicotinic acid hydrazide (1.37 g) to produce imine product L1. The aforementioned mixture was refluxed for 4 hours and then cooled to laboratory room temperature. The resulting product was filtered and vacuum-dried, yielding 82% pure chemical compound.

Synthesis of 1,3-benzodioxol-4-yl-methylene isonicotionohydrazide, compound L_2

The reflux method was used to synthesize Schiff base compound L2 by mixing 10 mmol of isonicotinic acid hydrazide (1.37 g) and 10 mmol of 1,3-benzodioxole-5-carboxaldehyde (1.5 mL) in 50 mL absolute ethanol with 3 drops of glacial acetic acid.[26] The identical method as for L1 was used, and 87% of the chemical component was pure. The synthesis reactions for compounds L1 and L2 is depicted in Scheme 1.



Scheme 1. The synthesis of imine compounds *N'*-[(1*Z*)-(2-hydroxyphenyl)methylene]isonicotinohydrazide L1 and *N'*-[(1*Z*)-1,3-benzodioxol-4-ylmethylene]isonicotinohydrazide L2

Compounds L1 and L2 polymerization

Synthesis of polymer of L1

For 10 minutes, a vessel reaction containing formaldehyde 37-41% (10 mmol) was heated to 40 °C. Following that, formaldehyde solution was combined with ethanol solution containing 30 mmol of compound L1. The mixture was then progressively combined with 10% sodium hydroxide dissolved in ethanol until the pH reached 9. The mixture was subjected to heating at a temperature of 60 °C for a duration of 3 hours. When the reaction time was up, the non-reacted components were removed from the round bottom. The mixture was washed with hot deionized water and vacuum-dried on a regular basis. It was discovered that the polymer yield was 78%.

Synthesis of polymer of L2

Polyurethane was created by combining excess methylene diphenyl diisocyanate (MDI) with polyol (polyester) for 30 minutes at room temperature with 1% trimethyl amine acting as a catalyst; this reaction led to terminate the isocyanate. To create a homogenous solution, the required amount of Schiff base L2 (30% based on polyol) was added to the mixture at 25 °C and allowed for 24 hours to complete the polymerization process. To remove the unreacted components, the foam was washed with hot deionized water, filtered, and dried at 70 °C. It was observed that the polymer yield was 70%.

The electrochemical investigation of ligands

Cyclic voltammetry was conducted using a DY2311 Potentiostat equipped with three cells. The electrochemical investigations utilized a platinum-saturated Ag/AgCl electrode as the reference electrode, and a glassy carbon electrode as the working electrode. Voltammetric measurements were conducted at room temperature using a DMF solution containing 0.1 M tetrabutylammonium perchlorate (TBAP) as the electrolyte.

Results and Discussion

Characterization of L1 and L2

The FTIR spectra of the synthetic compound L1 in the is shown in Figure 1.

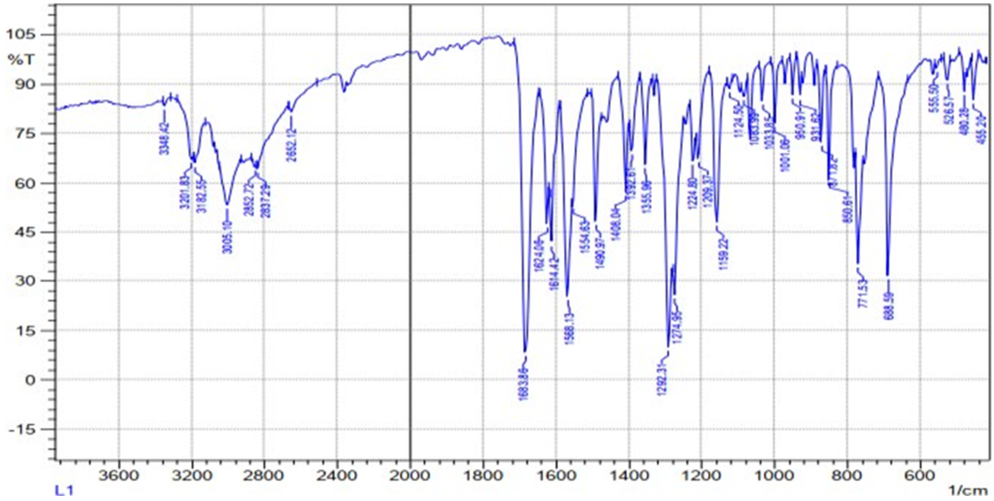


Figure 1. The FT- IR spectrum of compound L1.

There is strong evidence that the aldehyde and amine groups interacted to form an imine group (C=N) because there is no primary amine ($-NH_2$) band between 3300 and 3400 cm^{-1} or aldehyde band at ~2720 cm^{-1}. The existence of a robust stretching band at 1624 cm^{-1}, which is associated with the band (C=C), lends credence to this. The amide NH absorption peak was identified at 1685 cm^{-1} in both compounds. The C-O-C band for L2 is associated with an extremely prominent absorption peak between 1250 and 1050 cm^{-1}, whereas the O-H band for L1 is represented by the band at 3201 cm^{-1}. The other bundles' appearance is consistent with the literature, as shown in Figure 2.[27]

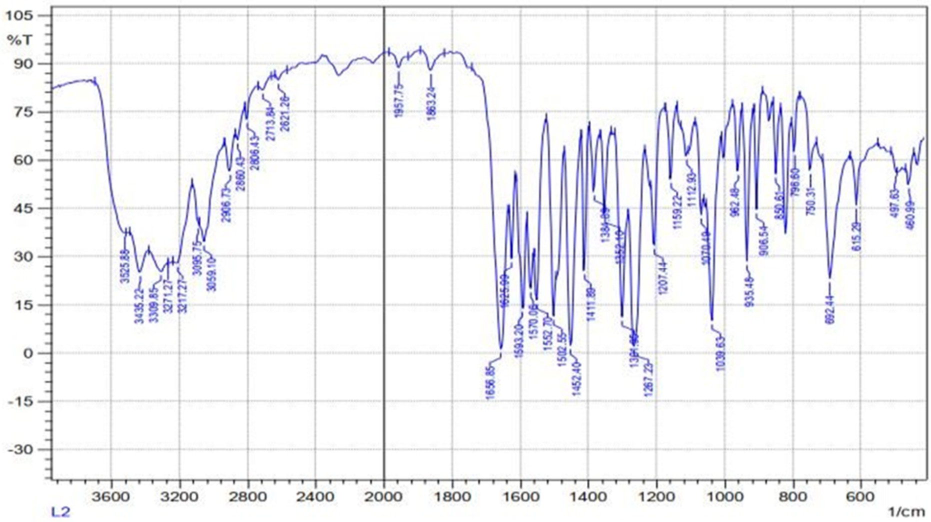


Figure 2. The FT- IR spectrum of compound L2.

The typical ^{1}H-NMR spectra of the produced compounds (imines) were obtained by using d6-DMSO at room temperature. The phenolic -OH proton is assigned a signal at 12.40 ppm in the ^{1}H-NMR spectrum of imine L1, while the amidic -NH- proton is denoted by a signal at 11.18 ppm. It is also possible to assign a singlet to the protons of pyridine rings at 8.90 ppm.[27] At 8.67 ppm, one imine proton was detected. Similarly, aromatic system proton locations are accurately predicted. At 6.91-7.85 ppm, the H-aromatic of the ligand imine 1 was detected,[28] Figure 3 shows this. Also, the amidic -NH proton in ligand 2's ^{1}H-NMR spectra is assigned a signal at 11.96 ppm. Pyridinium ring protons may also be indicated by a singlet at 8.72 ppm. A single proton of an imine was detected at 8.37 ppm. In addition, the aromatic protons of (imine L2) emerge at the expected locations. The ligand's aromatic protons were detected between 6.99 and 7.81 ppm.

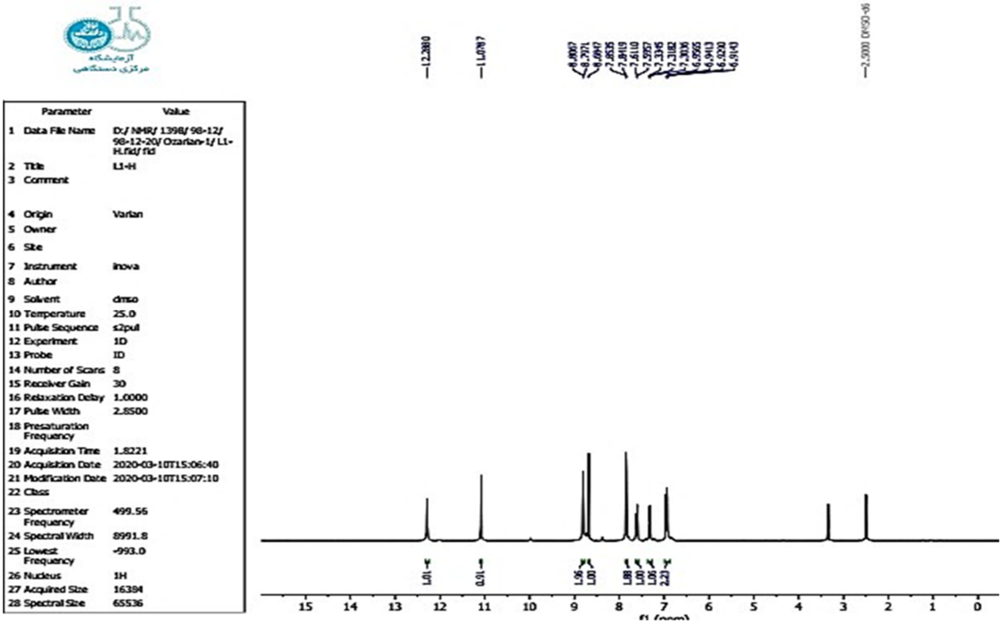


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Figure 3. ^{1}H-NMR spectrum of compound L1.

Furthermore, as indicated in Figure 4, methane protons CH_2-O were discovered at 6.10 ppm. Ion fragments from mass spectrometry were used to investigate the molecular structure of L1 and L2. Figure 4 shows that the mass spectrum for the L1 and L2 products, m/z 241 and 269, were similar to the ligands' molecular masses, with the base peak at m/z 78 and 147. The mass spectrum of L1 demonstrates that m/z 149 was formed via cleavage, resulting in the release of a $C_7H_7N_3O$ molecule.[29] The $C_6H_7N_2O$ molecule is responsible for another peak at m/z 123. The C_7H_7O molecule is assigned the molecular ion with m/z 106. Furthermore, cleavage produced the L2 component with m/z 163, releasing a $C_8H_7N_2O_2$ molecule. The $C_6H_7N_2O$ molecule is responsible for another peak at m/z 123.

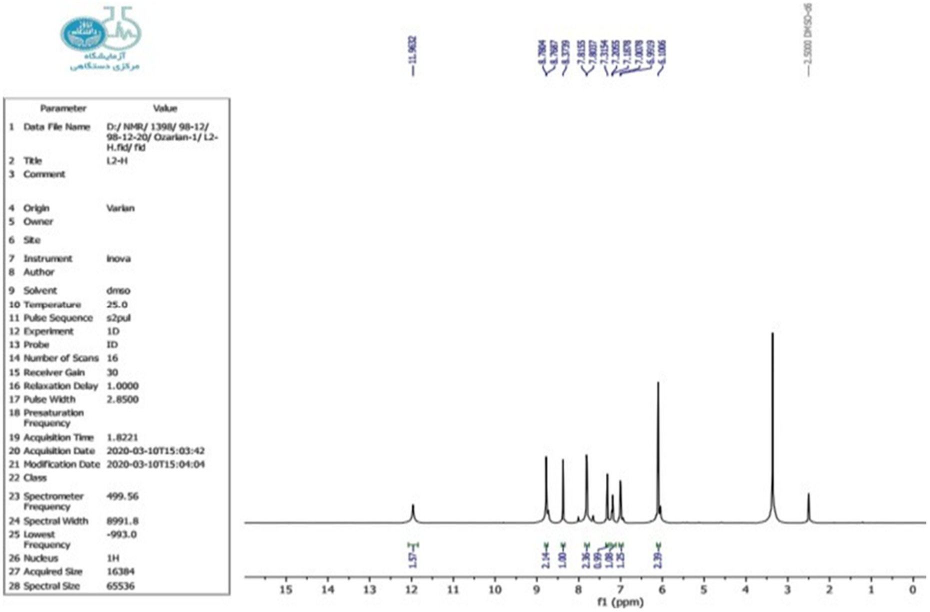

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Figure 4. ^{1}H-NMR spectrum of compound L2.

Figures 5 and 6 attribute the molecular ion with m/z 133 to the $C_7H_5N_2O$ molecule.[30]

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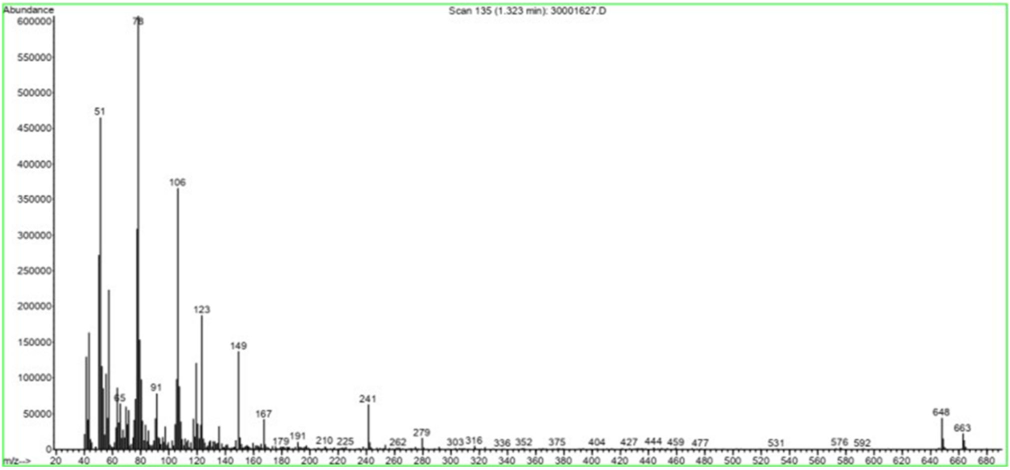

Figure 5. Mass spectra of compound L1.

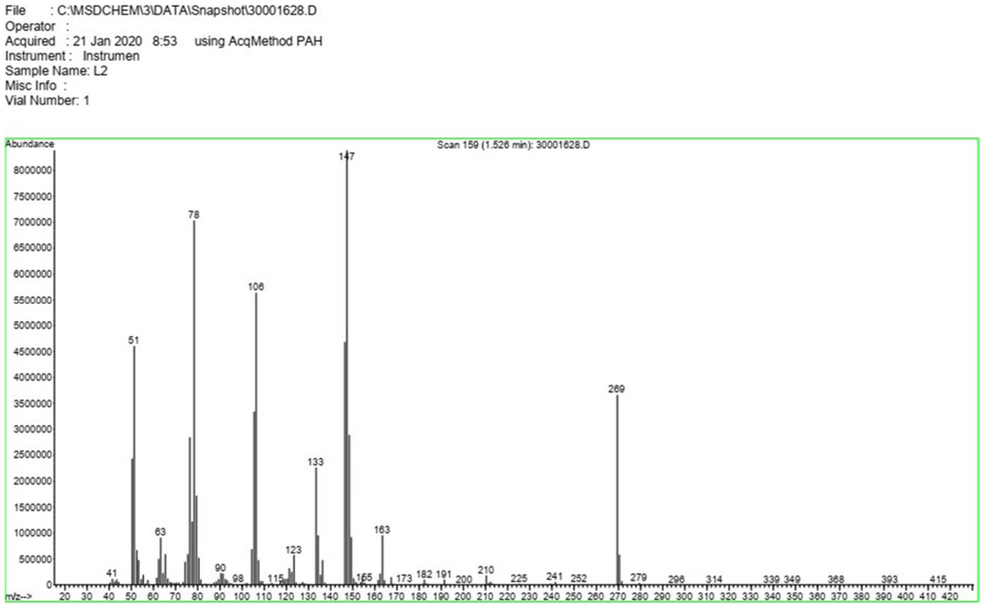

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Figure 6. Mass spectra of compound L2.

Thermogravimetric analysis (TGA)

The thermogravimetric analysis of polymer resins is shown in Figures 7 and 8.

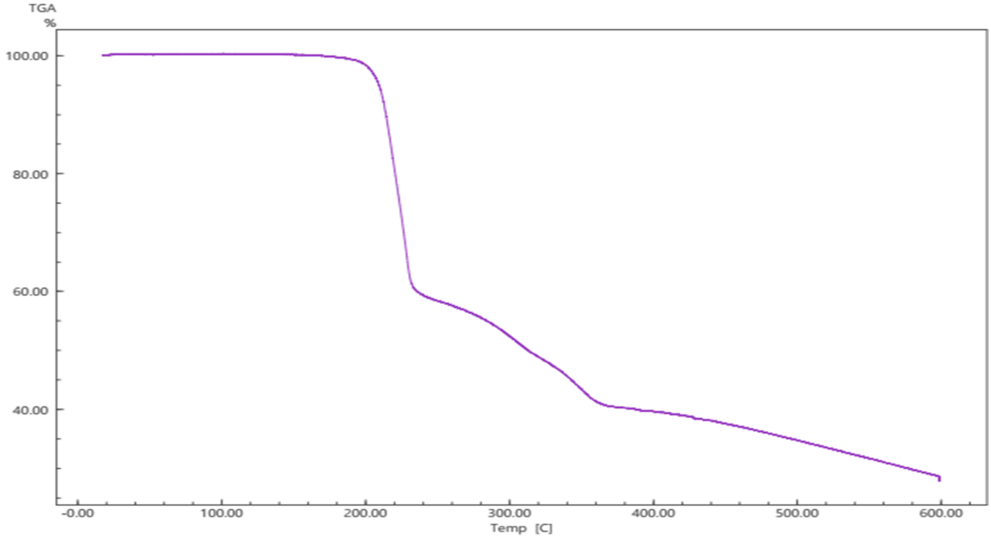

Figure 7. TGA curve of polymer resins of compound L1.

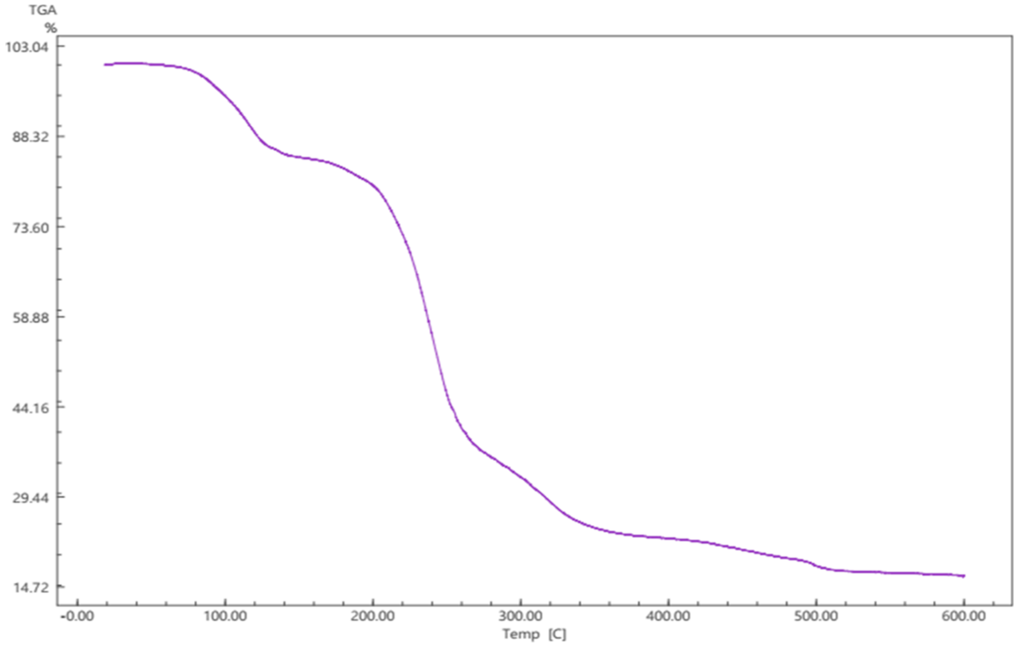

Figure 8. TGA curve of polymer resins of compound L2.

The graph clearly illustrates that L1 loses weight in two stages, with the first stage losing 40% of its weight at temperatures of 200°C. Furthermore, the second stage is 60% at temperatures of 365°C. Then, the L2 has two principal weight loss stages; the first stage offers weight losses of 15% at 105°C. Next, the second stage is 70% at temperatures of 295°C. The results indicate that L1 is more thermally stable than L2.[31]

Electrochemical study of the Schiff base

Cyclic voltammetry was conducted by performing two scans within the voltage range of +1.5 to -1.5 V. The experiments utilized 10 mM solutions of Schiff base in DMF, with 0.1 M tetrabutylammonium perchlorate (TBAP) serving as the supporting electrolyte. The scan speeds employed were 50 mV/s (Figure 9).

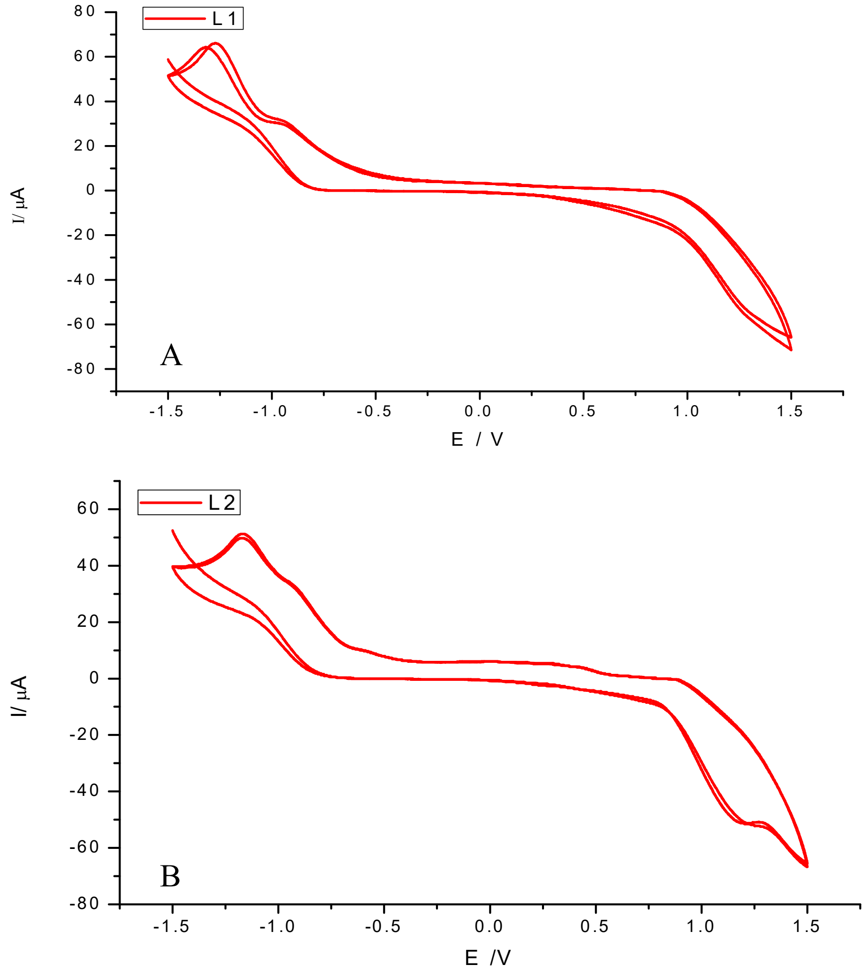

Figure 9. Cyclic voltammetry of Schiff base (10 mM) for compound A- L1 and B- L_2 in acetonitrile (0.10 M TBAP) with a scan rate of 50 mV/s, at 25 °C.

Figure 9A shows a typical cyclic voltammetry of L1, a Schiff base ligand. At Epc = -0.90 V and Epa = -0.85 V, cyclic voltammetry revealed one quasi-reversible event due to Schiff base ligand degradation. An anodic peak at Epa = -1.35 V was also found, which can be attributed to ligand OH oxidation. A cyclic voltammetry of Schiff base ligand L2 is also shown in Figure 9B. The L2 compound exhibits anodic and cathodic peaks at -1.15 V and -0.94 V, respectively. These peaks suggest a quasi-reversible redox mechanism resulting from the oxidation of the ligand. A second redox peak was observed in the curve as a result of a quasi-reversible process at Epa = +0.95 V and Epc = +1.15 V, attributed to the reduction of the Schiff base ligand.

Analytical study

A batch technique was used to assess the chelation efficiency qualities of L1 and L2 resins for numerous metal ions such as Zn^{2+}, Cu^{2+}, Fe^{2+}, Co^{2+}, and Ni^{2+}. Metal ions are removed from the ligands by mixing them with a 5 mL solution having 100 g/mL metal ion concentration. The pH was adjusted to an acceptable value (pH = 4) for each metal ion.[32] The impact of time on the maximum capacity under load was investigated by subjecting the samples to various treatment durations, ranging from 1 hour to 6 hours, while maintaining a constant pH level. This experimental setup is illustrated in Figure 9A and 9B. The loaded capacity for L1 increased in the following order under realistic conditions: Zn^{2+}, Cu^{2+}, Fe^{2+}, Co^{2+}, and Ni^{2+}. However, the order was different for Zn^{2+}, Cu^{2+}, Fe^{2+}, Co^{2+}, and Ni^{2+}. Ultimately, the metal solutions underwent filtration, and the remaining concentration of ions in the solutions was measured using an atomic absorption spectrophotometer.

Conclusion

The condensation procedure was utilized to manufacture two ligands in this work. The identification of these ligands was accomplished through a diverse range of methods, such as FT-IR, mass spectrometry, ^{1}H-NMR, TGA analysis, and electrochemical tests. Furthermore, two polymer resins were synthesized using L1 and L2 in order to assess the adsorption of metal ions in a water-based solution. The developed ligands have a high metal ion adsorption capability, with values ranging from Zn^{2+}, Cu^{2+}, Fe^{2+}, Co^{2+}, and Ni^{2+}.

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