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Green Synthesis, Structural Clarification, and Spectral Analysis of Schiff Base Ni(II) and Cu(II) Complexes Based on Pyridines with Associated Biological Significance

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Key words

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Abstract

This study used a solvent-free mechanochemical method as a green synthesis strategy to create two pyridine-based Schiff base ligands (H_2L_1 and H_2L_2) produced from 2,3-dihydroxybenzaldehyde. Mechanochemical grinding and microwave assistance were used to create their equivalent nickel(II) and copper(II) complexes. Using elemental analysis, FT-IR, mass spectrometry, and molar conductivity tests, the ligands and complexes were thoroughly described. Depending on the ligand structure, spectroscopic data verified ON and ONN coordination modes, resulting in octahedral and distorted octahedral geometries surrounding the metal centers. In view of their possible biological ramifications, structural characteristics were examined, laying the groundwork for further anticancer assessment.

I. Introduction

Because of their easy synthesis, flexible coordination behavior, and potent metal-binding capacity through azomethine nitrogen and extra donor atoms, schiff base ligands play a crucial role in coordination chemistry. Heterocyclic moieties like pyridine promote electronic delocalization and chelation strength, producing complexes with better stability and functional characteristics (Cozzi, 2004)(Caracelli, Haiduc, Zukerman-Schpector, & Tiekink, 2014)

Due to their varied coordination geometries and importance in catalysis, materials science, and medicinal chemistry, nickel(II) and copper(II) Schiff base complexes have been thoroughly studied (Nakamoto, 2009) (Bruno, De Robertis, Giuffrè, Rotondo, & Sammartano, 2009; Lever & Rice, 1969). Cu(II) complexes in particular have garnered a lot of interest due to their biological significance and redox activity.The application of green chemistry principles has grown in significance in tandem with these advancements.The application of green chemistry principles has grown in significance in tandem with these advancements. With shorter reaction durations, less solvent use, and higher yields, mechanochemical and microwave-assisted syntheses are safe substitutes for traditional solvent-based methods (Kumar et al., 2010) (James et al., 2012).The current study combines comprehensive structural and spectroscopic characterization of novel pyridine-based Schiff base Ni(II) and Cu(II) complexes with environmentally friendly synthesis techniques.

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II. Experimental

2.1Instrumentation Used

The following instruments were used for the spectroscopic and physical measurements of the prepared ligands and their complexes:

-1 Melting Point Measurement

The melting point of the prepared ligands was measured using a Stuart Point Melting Instrument (SM10).

-2 Infrared Spectroscopy

The infrared spectra of the prepared ligands were recorded using a Perkin FT-IR Spectrometer (Elmer FT-IR Spectrometer, USA, 1 cm^{-1}, 4000 nm).

-3 Elemental Analysis

The carbon and nitrogen content was measured. Hydrogen (C.H.N) for the ligands.

-5 Mass Spectrometry

The mass spectra of the prepared ligands were recorded using an autospec Micromass spectrometer.

-6 Nuclear Magnetic Resonance Spectroscopy

The proton 1HNMR NMR spectra of the prepared ligands were measured using a Bruker Avance III NMR spectrometer as a reference.

2.2 Preparation of Ligands

2.2.1 -N-(2,3-dihydroxybenzylidine)-pyridine-2-amine(H2L1) ligand preparation:

By mixing this ligand, 0.94 g of 2,3-dihydroxybenzaldehyde (10 mmol, 1.38 g) and 2,3-dihydroxybenzaldehyde (10 mmol, 1.38 g) were prepared.

By mixing this ligand, 0.94 g of 2,3-aminopyridine (10 mmol) were ground together in a mortar and pestle for 10 minutes. A dark red solid was obtained. The formation of the ligand was confirmed using TLC.

2.2.2-N-(2,3-dihydroxybenzylidine)-pyridine-3-amine(H2L2) ligand preparation:

By mixing this ligand, 0.94 g of 2,3-dihydroxybenzaldehyde (10 mmol, 1.38 g) and 2,3-dihydroxybenzaldehyde (10 mmol, 1.38 g) were prepared. Combined in a mortar and pestle, the materials were thoroughly ground.

After 8 minutes, an orange solid was obtained. The formation of the ligand was confirmed using TLC.

Results and discussion

1.3 Preparation

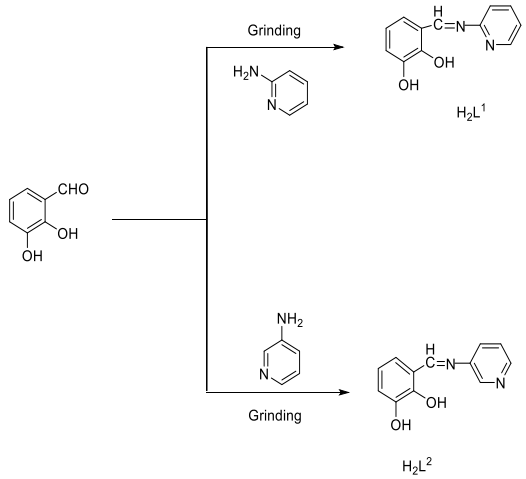
1.1.3 Preparation of Schiff Base Ligands

In this study, Schiff bases

N-(2,3-dihydroxybenzylidine)-pyridine-2-amine (H_2L_1) and

N-(2,3-dihydroxybenzylidine)-pyridine-3-amine (H_2L_2) were prepared using mechanical grinding as a simple and environmentally friendly green preparation method.

The ligands H_2L_1 and H_2L_2 were 2-aminopyridine in the ligand state, with 2,3-dihydroxybenzaldehyde in the ligand state, and 3-aminopyridine in the ligand state. The structures of these ligands were confirmed using elemental analysis, mass spectrometry, infrared spectroscopy, and nuclear magnetic resonance spectroscopy. The analytical and spectroscopic data are consistent with previously prepared similar compounds(El-Ajaily, Al-Barki, & Maihub, 2016).Scheme (3-.1) illustrates the preparation of the ligands.



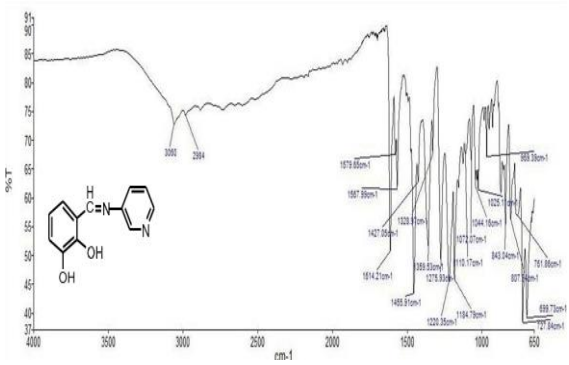
Scheme (3-1): Preparation of ligands H_2L_1 and H_2L_2

2.3 Infrared Spectra

1.2.3 Infrared Spectra of H2L1 Ligand and its Complexes

Figure (3-1) shows the infrared spectrum of H_2L_1 ligand. An absorption band at 1620 cm^{-1} is attributed to the adsorption of the azomethine (CH=N) group in H_2L_1 ligand.(Aliyu & Ado, 2010).Additionally, the band at 1557 cm^{-1} is attributed to the adsorption of the (C=N) pyridine group in H_2L_1 ligand.

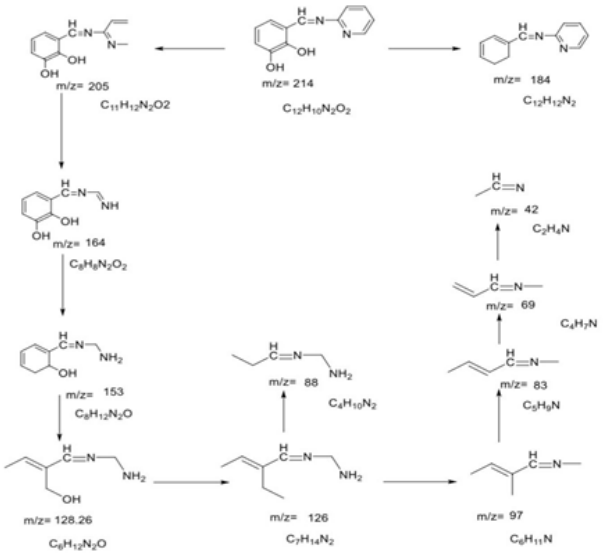
The absorption band at 1367 cm^{-1} is attributed to the adsorption of the phenolic C–O group in the ligand. (Lever & Rice, 1969) (İnci, Aydın, & Zorlu, 2021)



H2L1 Figure (3-1): Infrared spectrum of H_2L_1 ligand

2.2.3 Infrared Spectra of H_2L_2 Ligand and its Complexes

The infrared spectrum of H_2L_2 ligand is shown in Figure (3-2). The proposed subdivisions for the first ligand are shown in the Scheme(3-2).



Scheme (3-2): Mass fractions of H2L1 ligand

In the ligand's spectrum, we observe an absorption band at 1614 cm^{-1}, which is attributed to the adsorption of the azomethine group (CH=N), and a band at 1567 cm^{-1}, which is attributed to the adsorption of the pyridine group (C=N) (Lever & Rice, 1969). The band at 1359 cm^{-1} is attributed to the adsorption of the phenolic C–O group in H2L2 ligand (Zhu et al., 2013).

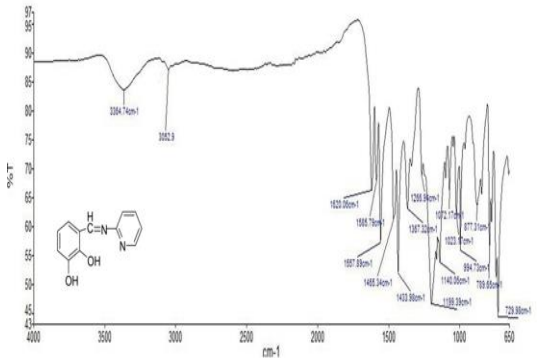


Figure (3-2) Infrared spectrum of H_2L_2 ligand

Based on the above, it can be predicted that the Schiff base ligand (H_2L_1), derived from 2-aminopyridine, is a tridentate glycoside linked to copper(II) and nickel(II) ions via the azomethine nitrogen atom, the phenolic oxygen atom, and the pyridine ring nitrogen atom. Meanwhile, the ligand (H_2L_2), derived from 3-aminopyridine, is a bidentate glycoside linked via the azomethine group nitrogen and the phenolic oxygen atom.

3.3 Mass Spectra

1.3.3 Mass Spectrum of the First Ligand H2L1

The first ligand yielded a set of peaks, as shown in Figure '(3-4)

The mass spectrum of the first ligand was recorded.

The proposed partitions for the first ligand are shown in Scheme (3-3). The figure shows a major peak at m/z+ = 214, corresponding to the molecular weight of the ligand C12H10N2O2, whose molecular weight of 214 supports the proposed structure of the H2L1 ligand, as the mass spectrum showed a peak at m/z+=126 It goes

back to .C7H14N2.

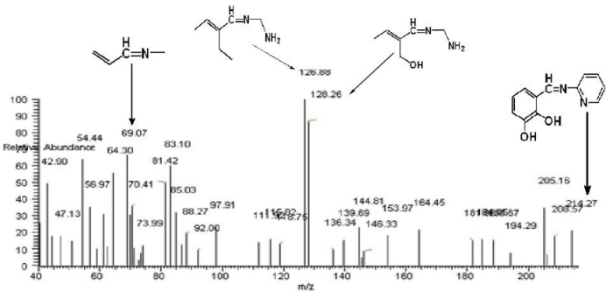
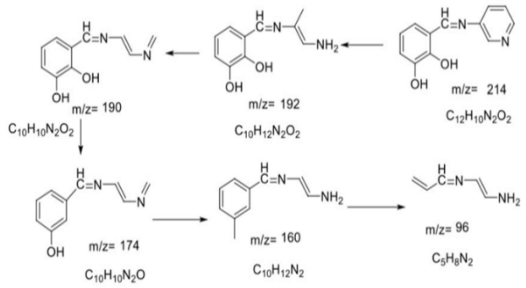


Figure (3-3): Mass spectrum of H2L1 ligand

Mass Spectrum of H2L2 Ligand

The mass spectrum of H2L2 ligand was recorded, yielding a series of peaks as shown in Figure (3-4). The proposed fractions for the first ligand are shown in Scheme (3-3).



Scheme (3-3): Proposed fractions for H2L2 ligand

The figure shows a major peak at 214 m/z corresponding to the molecular weight of the ligand C12H10N2O2, whose molecular weight of 214 supports the proposed structure of H2L2 ligand. The mass spectrum also showed a peak at 190 m/z corresponding to C10H10N2O2, a peak at 96 m/z corresponding to C5H8N2, and a peak at 74 m/z corresponding to C4H11O.

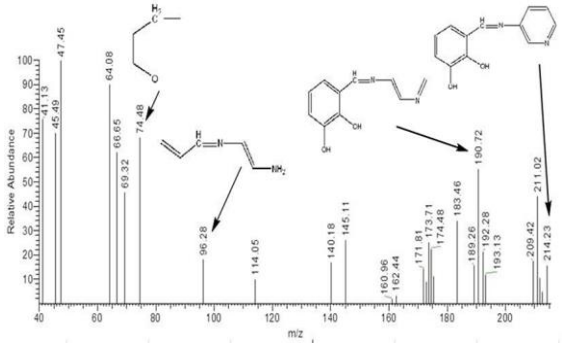


Figure (3-4): Mass spectrum of H2L2 ligand

1HNMR Spectra

The nuclear magnetic resonance (NMR) spectra of H2L1 and H2L2 ligands were studied using DMSO-d6 and TMS as a standard reference. The 1HNMR spectrum of ligand H2L1 (Figure 3-5) and ligand H2L2 (Figure 3-6) showed two chemical shift bands of 9.44 (10.20 ppm) in H2L1 and 8.97 (10.20 ppm) in H2L2, representing the protons of the phenolic OH group (Ünver, Yıldız, Kiraz, & Özgen, 2009). The spectrum also showed a chemical shift signal of 8.53 ppm and 8.63 ppm in ligands H2L1 and H2L2, respectively, attributed to the azomethine group proton (Odularu, 2023). The aromatic proton absorption region appeared in the 7.92–6.45 ppm region, attributed to the protons in the two aromatic rings (Abdel-Rahman, El-Khatib, Nassr, & Abu-Dief, 2017).

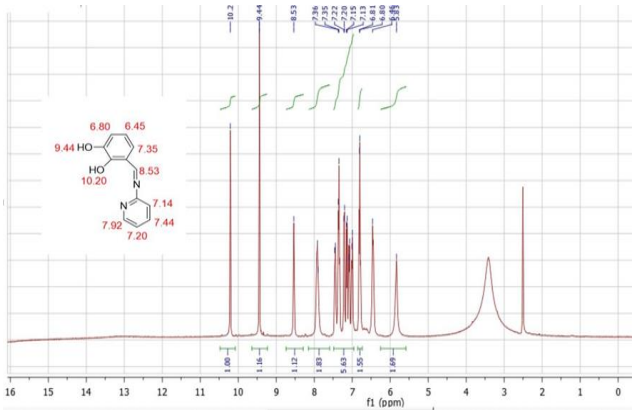


Figure (3-5) shows the nuclear magnetic resonance spectrum of the H2L1 ligand.

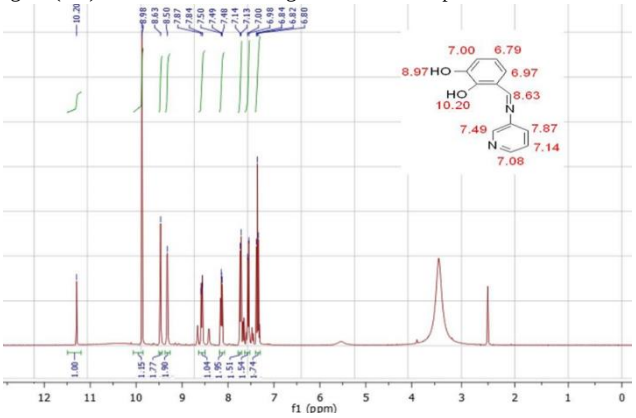


Figure (3-6) shows the nuclear magnetic resonance spectrum of the H2L2 ligand.

conclusion

The effective synthesis of structurally well-defined Schiff base Ni(II) and Cu(II) complexes based on pyridines was made possible via green synthetic methods. A strong structural framework for understanding their biological performance is provided by the clarified coordination modalities and geometries.

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التخليق الأخضر، والتوضيح البنيوي، والتحليل الطيفي لمركبات شيف القاعدية Ni(II) و Cu(II) القائمة على البيريدينات ذات الأهمية البيولوجية المرتبطة بها

فايزة فرج إسويسي، هنا بشير شاويش،
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استلمت الورقة بتاريخ 2026/01/03، وقبلت بتاريخ 2026/01/14، ونشرت بتاريخ 2026/01/15

الكلمات المفتاحية: معقدات قواعد شيف، وروابط البيريدين، والتخليق الأخضر، والنيكل (II)، والنحاس (II)، والخصائص الطيفية

الملخص

استخدمت هذه الدراسة طريقة ميكانيكية كيميائية خالية من المذيبات كاستراتيجية تصنيع صديقة للبيئة لإنتاج اثنين من روابط شيف القاعدية القائمة على البيريدين (H_2L_1 وH_2L_2) المُنتَجة من 2،3-ثنائي هيدروكسي بنزالدهيد. استُخدم الطحن الميكانيكي الكيميائي والمساعدة بالميكروويف لإنتاج مُركّبات النيكل (**II**) والنحاس (**II**) المُكافئة لها. وباستخدام التحليل العنصري، وقياس طيف الأشعة تحت الحمراء بتحويل فورييه (**FT-IR**)، وقياس الطيف الكتلي، واختبارات الموصلية المولية، وُصِفت الروابط والمُركّبات وصفًا دقيقًا. وبناءً على بنية الرابطة، أكّدت البيانات الطيفية أنماط التناسق **ON** و**ONN**، مما أدّى إلى تكوين أشكال هندسية ثمانية السطوح وثمانية السطوح مُشوّهة تُحيط بالمراكز المعدنية. ونظرًا لتداعياتها البيولوجية المُحتملة، دُرست الخصائص البنيوية، مما يُمهّد الطريق لمزيد من التقييم المُضاد للسرطان.