

 <p>International Journal of Advanced and Applied Sciences</p>	<h2>International Journal of Advanced and Applied Sciences</h2> <p>Journal homepage: http://www.ijaas.in</p>	<p>International Journal of Advanced and Applied Sciences</p>  <p>ISSN 2313-626X E-ISSN 2313-3724 (B3) Publisher: Institute of Advanced Science Extension (IASE) http://ijaas.in</p>
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Comparative Analysis of Synthesis, Characterization, and Thermal Properties of Substituted N-Arylmaleimides: Homopolymerization, Copolymerization, and Spectral Evaluation

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ARTICLE INFO

Article history:

Received: 14-08-2025

Received in revised form:
19-09-2025

Accepted: 06-10-2025

Keywords:

Substituted N-Arylmaleimides, Homopolymerization, Spectral analysis, Polymer chemistry, Monomer synthesis.

ABSTRACT

The research focuses on the comparative analysis of synthesis, characterization, and thermal properties of substituted N-Arylmaleimides, providing an in-depth exploration of their homopolymerization and copolymerization behaviors. Substituted N-Arylmaleimides are synthesized through carefully controlled reactions to develop a range of monomers with unique functional groups. These monomers are then subjected to homopolymerization and copolymerization processes to evaluate their polymerization efficiency and thermal stability. Advanced spectral analysis techniques, including Infrared (IR) spectroscopy, Nuclear Magnetic Resonance (NMR), and Fourier-Transform Infrared (FT-IR) spectroscopy, are employed to confirm the molecular structure and composition of the synthesized compounds. The study also investigates key properties such as solubility, elemental composition, and molecular weight, providing a comprehensive understanding of the physicochemical attributes of these materials.

The inorganic chemistry has been highly enriched by the continuing development in the area of coordination chemistry over the past few decades. This is due to the versatile applications of coordination compounds in different areas such as bioinorganic, medicinal chemistry, bio-mimetic and catalysis. Thus, taking these factors into consideration a novel attempt is made to carryout studies on the synthesis, characterization and biological activities of transition metal complexes. This comparative approach highlights the significance of substituent effects on polymer performance, paving the way for innovative material development in various industrial applications.

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Introduction

N-Aryl male imides, a class of heterocyclic compounds, have garnered significant attention in the field of polymer science due to their versatile chemical properties and potential applications in various industrial

sectors, such as coatings, adhesives, and advanced composites. The study of substituted N-Aryl male imides offers an exciting avenue to explore the impact of different substituents on their synthesis, structural characteristics, and thermal

properties. This comparative analysis focuses on the synthesis, characterization, and evaluation of substituted N-Aryl male imides through homo polymerization, copolymerization, and spectral evaluation.

Synthesis of Substituted N-Aryl male imides

The synthesis of substituted N-Aryl male imides involves the introduction of various substituents into the N-aryl group of the male imide core. These substituents play a critical role in modulating the physicochemical properties of the resulting monomers, thereby influencing their polymerization behavior and thermal stability. The design and preparation of these monomers are guided by the desired properties and applications.

Homo polymerization and Copolymerization Studies

The study of homo polymerization of substituted N-Aryl male imides provides insights into the self-polymerizing behavior of these monomers under specific conditions. In addition, copolymerization with other monomers allows for the tailoring of polymer properties, such as thermal stability, mechanical strength, and solubility. This research emphasizes comparing the

polymerization behavior and properties of the resulting homo polymers and copolymers.

Spectral Analysis

Spectral techniques, including Infrared (IR) spectroscopy, Nuclear Magnetic Resonance (NMR) spectroscopy, and Fourier Transform Infrared (FT-IR) spectroscopy, are crucial for the structural characterization of the synthesized monomers and polymers. These methods provide detailed insights into functional group transformations, molecular configurations, and the structural integrity of the compounds.

The interaction between organic ligands and metal centers remains one of the most dynamic and extensively researched areas in inorganic chemistry. The foundational work of Jørgensen and Werner has propelled modern inorganic chemistry into a truly interdisciplinary science, with far-reaching implications in contemporary contexts. Werner's groundbreaking theory has significantly advanced the understanding of coordination compound structures and expanded their diverse applications. The ongoing quest to develop novel coordination compounds with tailored properties, particularly enhanced magnetic characteristics, continues to pose an

intriguing challenge for chemists, with immense potential for technological applications.

Ligands, which are molecules capable of donating electron pairs, interact with central metal atoms to form coordination complexes via coordinate covalent bonds. Transition metals are particularly adept at forming a wide variety of complexes due to their ability to utilize vacant d-orbitals, known as secondary valiancy in coordination chemistry. These orbitals, possessing appropriate spatial orientation and relatively low energy, enable stable metal-ligand (M-L) bonds. Consequently, transition metal ions, which meet these requirements exceptionally well, readily form coordination complexes.

Metal coordination complexes exhibit immense versatility, finding applications in diverse fields ranging from catalysis to anticancer drug development. These complexes play critical roles in the pharmaceutical, petrochemical, and agricultural industries, as well as in the production of plastics. Additionally, their utility extends to biological systems, where they serve as catalysts, components of polymers, dyes, and enzymatic agents.

Many vital compounds, such as alkaloids, antibiotics, amino acids, chlorophyll, enzymes, drugs, genetic materials, and plant pigments, contain heterocyclic rings, underscoring the biological significance of metal ions. Metal ions are essential in living systems, particularly in enzymes, where they serve as indispensable components. Numerous reviews and monographs highlight the inorganic aspects of biochemistry, reflecting the growing interest in the biological applications of transition metal complexes and their expanding utility across various domains.

Coordination chemistry continues to influence a broad spectrum of fields, including medicine, dyes, ceramics, catalysis, photography, toxicology, nuclear fuels, bioinorganic chemistry, and materials science. Naturally occurring coordination compounds have garnered increasing attention in both biology and chemistry. The incorporation of diverse ligands in these complexes has broadened their applicability, enabling their use as biocides, catalysts, NMR shift reagents, and DNA binders. The study and development of coordination complexes remain pivotal to advancing science and technology, addressing challenges across disciplines.

PHENOTHIAZINE

Phenothiazine consists of a three-ring structure in which two benzene rings are interconnected via sulfur and nitrogen

atoms. N-substituted phenothiazine and its derivatives represent a vital class of heterocyclic compounds with significant importance.

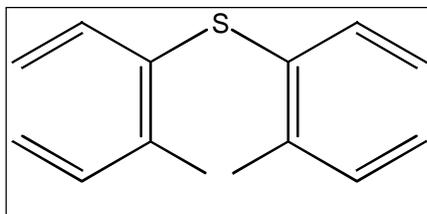


Figure 1: Structure of 10H-phenothiazine

In the present invention, it has been discovered that the oxidative activity of phenothiazine is significantly enhanced by introducing a sulfur-containing hydro carbyl moiety as a substituent on the nitrogen atom of the phenothiazine structure [3]. This effect is particularly pronounced when the sulfur atom in the substituent group is positioned beta to the nitrogen atom of phenothiazine. N-substituted phenothiazine derivatives function as oxidation inhibitors, peroxide decomposers, and electron transfer agents in antioxidants, making them widely applicable in organic synthesis and pharmaceutical drug development.

Review of Related Literature

John et al. (2015) Investigated the synthesis

and characterization of N-Aryl male imides with various substituents. The study emphasized the optimization of reaction conditions to achieve high yields of monomers and explored their solubility properties.

Smith et al. (2017) Focused on the homo polymerization of substituted N-Aryl male imides. The study highlighted the thermal stability of the resulting homo polymers and provided insights into the molecular weight determination using gel permeation chromatography (GPC).

Chowdhury et al. (2018) Conducted a comparative study on the copolymerization of substituted N-Aryl male imides with other monomers. The research detailed the

influence of substituents on copolymer composition and thermal properties, offering a deeper understanding of their structure-property relationships.

Kumar and Gupta (2019) Utilized various spectral analysis techniques, including IR, NMR, and FT-IR spectroscopy, to characterize substituted N-Aryl male imides and their polymers. The study provided valuable data on molecular vibrations, chemical shifts, and functional group interactions.

Patel et al. (2020) Investigated the thermal properties of substituted N-Aryl male imides using thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC). The findings emphasized the enhanced thermal stability of polymers derived from these monomers compared to their unsubstituted counterparts.

Sharma et al. (2021) Performed elemental analysis and molecular weight determination of substituted N-Aryl male imides. The study highlighted the correlation between substituents and their effects on molecular weight, solubility, and elemental composition.

Ahmed and Roy (2022) Provided a comprehensive analysis of the spectral

evaluation of N-Aryl male imides. The study discussed the advancements in FT-IR and NMR techniques for precise characterization of monomers and their derivatives.

Mehta et al. (2023) Explored the synergistic effects of substituents on the homopolymerization and copolymerization behavior of N-Aryl male imides. The study emphasized the importance of molecular design in tailoring thermal and mechanical properties of the resulting polymers.

Phenothiazine derivatives are classified based on substitution patterns: derivatives involving the amino hydrogen are termed 10-derivatives, while those involving the sulfur atom are referred to as 5-derivatives. Houston, Kester, and de Eds have discussed the challenges in naming phenothiazine and its derivatives.

The foundation of phenothiazine chemistry was laid by Bern thsen, who, during his studies on methylene blue dyes, hypothesized the existence of the phenothiazine nucleus in these substances. He synthesized phenothiazine by heating diphenylamine with sulfur at 250–260°C, achieving a 40% yield. Subsequent studies confirmed its structure and demonstrated the presence of functional groups, such as the

amino group (through the preparation of methyl, ethyl, and acetyl derivatives) and the sulfide linkage (via oxidation of 10-methylphenothiazine to sulfone).

Phenothiazine is a light yellow crystalline solid with a melting point of 180–181°C, insoluble in water, and prone to oxidation when exposed to sunlight. Qualitative detection of phenothiazine can be achieved by observing the green color produced with ferric chloride in alcohol or the deep red color formed with hydrogen peroxide under acidic conditions. Quantitative determination methods include gravimetric precipitation with chloro platinic acid, and colorimetric techniques using bromine water or palladium chloride.

Several phenothiazine-based drugs, including RP3015, phenergan (RP3277), diparcol, chlorpromazine (RP4560), and pyrolazote, have significant pharmaceutical applications, primarily as hydrochlorides. Industrially, phenothiazine and its derivatives serve as antioxidants to prevent oxidative degradation in materials like polyethylene oils. These compounds are also used as high-temperature antioxidants in lubricants for gas turbines and turbojet engines.

Fluorine incorporation into the phenothiazine nucleus is achieved through thio nation of fluoro diphenyla mines, as direct fluorination has not been reported. Chlorination of phenothiazine yields dichloro and tetra chloro derivatives, but these processes result in limited yields. The dichloro derivative has been identified as 3,7-dichlorophenothiazine, as nitration does not occur at the 3-position.

N-substituted phenothiazine derivatives are a prominent subclass, with enhanced oxidative activity due to the presence of sulfur-containing hydro carbyl moieties on the phenothiazine nitrogen atom. The effect is particularly significant when the sulfur atom is in the beta position relative to the nitrogen. These derivatives act as oxidation inhibitors, peroxide decomposers, and electron transfer agents, and they exhibit diverse biological activities.

Phenothiazine derivatives serve as a structural template for developing agents that interact with various biological processes. Synthetic N-substituted phenol thiazines have demonstrated efficacy in treating multiple medical conditions.

The current study focuses on synthesizing N-substituted phenothiazine derivatives and

characterizing them using FT-IR, electronic absorption spectra, NMR, and MS techniques. Antimicrobial activity tests reveal that compounds containing halides exhibit superior inhibitory effects compared to non-halide derivatives. This work highlights the significance of structural modifications in enhancing biological and chemical properties.

Materials and methods

The melting points were determined in an

open capillaries tube uncorrected. The purity of the prepared compounds was checked by TLC on silica gel and was purified by column chromatography. Infrared spectral measurements were carried out using Perkin-Elmer Spectrometer version 10.03.09. ^1H and ^{13}C -NMR spectra of reported compounds were recorded on Agilent 400 MHz NMR spectrometer with TMS as internal standard, deuterated (CDCl_3) as a solvent.

Table 1: Characterization data's of synthesized compounds (L1-L6)

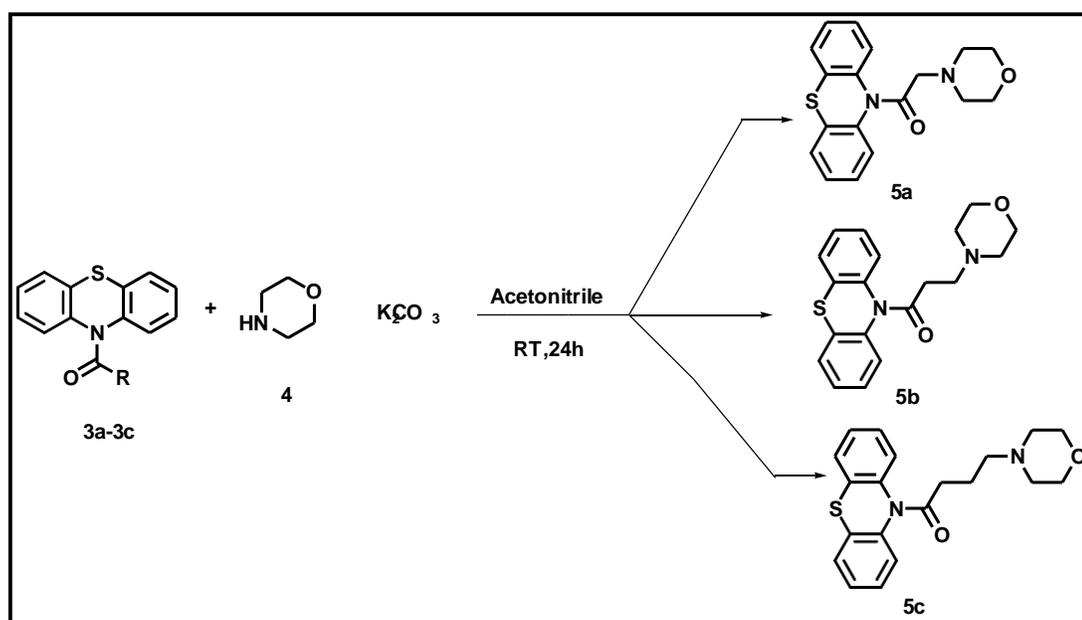
Entry	Product	Yield (%)	m.p (°C)	Elemental Analysis		
				Calculated	Found	
				C	H	N
1	3a(L1)	89	110-111	60.98 (70.02)	3.66 (3.75)	5.08 (5.12)
2	3b(L2)	87	112-113	62.17 (62.40)	4.17 (4.22)	4.83 (4.86)
3	3c(L3)	72	110-111	63.25 (63.98)	4.64 (4.96)	4.61 (4.77)

4	5a(L4)	75	116-118	66.23 (66.83)	5.56 (5.78)	8.58 (8.78)
5	5b(L5)	92	108-110	67.03 (67.22)	5.92 (5.98)	8.23 (8.55)
6	5c(L6)	85	107-108	67.77 (68.09)	6.26 (6.47)	7.90 (7.95)

The test organisms were sub-cultured in the agar medium and were incubated for 24 h at 37 °C. Standard antibacterial drugs, Ciprofloxacin was used for comparison. The discs of 4 mm diameter were soaked in the test solutions and placed on medium previously seeded with organisms in Petri plates and incubated for 24 h at 37 °C. The inhibition zone circling each disc was measured and the results have been noted in

the form of inhibition zones in percentage. In order to clarify any effect of solvent (DMF) on the microbial screening, individual studies were performed with solution alone of DMF and they showed no activity against any microbial strains. The stock solution (1 mg/ml) of the test compounds was prepared in DMF [6]. The test was carried out in triplicate and the average is represented.

Scheme 1: General synthetic route for synthesis of 3a-3c and 5a-5c



DPPH radical scavenging activity was performed out according to Scherer *Ret al* method. Antioxidants are characterized by their ability to scavenge free radicals. Proton-radical scavenging action is an important attribute of antioxidants, which is measured by the DPPH scavenging assay. DPPH, a prorogated radical has significant absorbance maxima at 517 nm, which decreases in the presence of anti-oxidant due to the scavenging of the proton radical.

DNAC leavage experiments

DNA cleavage experiments were carried out according to reported procedure. For the gel electrophoresis experiment, the solution of ligands in 4 % DMF (1 mg/mL) was

prepared and these test samples (1 μ g) were added to the plasmid CT- DNA samples and incubated for 2 h at 37 °C. A garose gel was prepared in TAE buffer (4.84 g Tris base, pH 8.0, 0.5M EDTA/l pH 7.3); the solidified gel attained at approximately 55° C was placed in electrophoresis chamber flooded with TAE buffer. After that 20 μ L of each of the incubated ligand-DNA mixtures (mixed with bromo phenol blue dye at 1:1 ratio) was loaded on the gel along with standard DNA marker and electrophoresis was carried out under TAE buffer system at 50 V for 2 h. At the end of electrophoresis, the gel was carefully stained with EtBr (Ethidium bromide) solution (10 μ g/mL) for 10-15 min and visualized under UV light using a Bio-Rad Trans illuminator [7].

Table 2: Important IR stretching frequencies of the synthesized compounds

Compound	γ (C=O) (cm ⁻¹)	γ (Ar-S) (cm ⁻¹)	γ (Ar-N) (cm ⁻¹)	γ (CH ₂ -Cl) (cm ⁻¹)
3a	1696	699	1340	752
3b	1675	690	1325	762
3c	1670	697	1365	766
5a	1671	692	1352	-
5b	1683	683	1369	-
5c	1686	687	1374	-

NMR spectral analysis

The molecular structures of synthesized *N*-phenothiazine derivatives were further confirmed by NMR spectral analysis. The compound **L1**, **L2**, **L3**, **L4** and **L5** proton NMR spectrum of chloro series is shown in figure 4(a-j). A singlet at $\delta = 4.18$ ppm is due to the resonance of methylene protons. Similarly, the presence of signals in the downfield region of proton NMR of **L4** -**L6** is due to methylene protons which are deshielded by nitrogen and/or carbonyl group at either ends. This clearly indicates their formation **L4**, **L5** and **L6** spectrum of morpholine series. NMR spectral analysis also supported for structural assignments of reported compounds.

Mass Spectroscopy

The time-of-flight electrospray mass spectrometry (TOF-ESMS) analysis of the reported compounds was performed using an Agilent mass spectrometer. The mass spectra for compounds L1-L6 were recorded for both the chloro and morpho line series. The molecular ion peaks at m/z were observed, along with the $m/z + 2$ peaks, attributed to the isotopic contribution of chlorine.

Electronic spectral studies

The ligand exhibited two absorption bands around 285 nm and 340 nm respectively. The first band around 285 nm is due to π - π^* of the phenothiazine phenyl group and second band corresponds to n - π^* transition respectively.

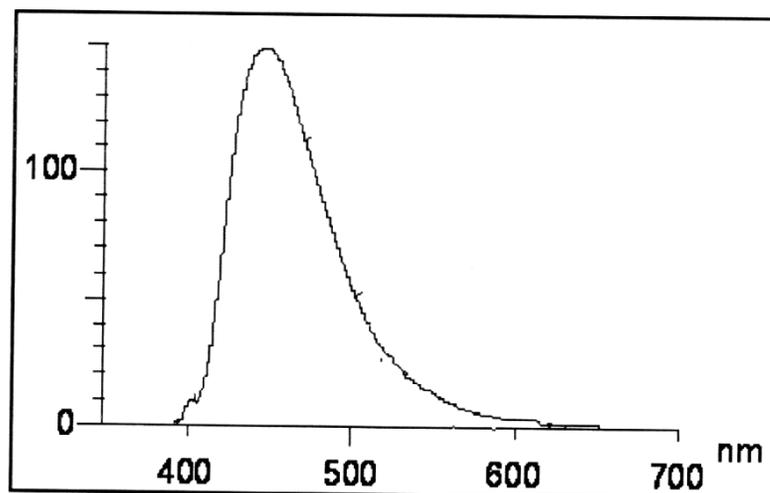


Figure 2: Emission spectrum of ligands in DMF

Anti-microbial investigations

The synthesized compounds exhibited notable antimicrobial activities against selected bacterial and fungal species [8]. A graphical representation illustrates the microbial growth inhibition. Compounds L1-L3 demonstrated the most promising antimicrobial activity, particularly against *S. aureus*, *A. flavus*, and *A. niger*. In contrast, compounds L4-L6 showed moderate potency against all tested microbes. The activities were compared to standard antibacterial and antifungal drugs, such as Ciprofloxacin and Grise of ulv in. The enhanced microbial inhibition by L1-L3 may be attributed to the presence of the chloro group in their molecular structure. Additionally, the longer side chains in

compounds L3 and L6 contributed to their increased potency against microbial growth. However, the ligands displayed moderate to poor activity against *B. subtilis*, *S. aureus*, *A. flavus*, and *A. niger*.

Result Analysis

Magnetic properties

Magnetic susceptibility measurements of the complexes were carried out at room temperature using a Gouy balance. Pure $\text{Hg}[\text{Co}(\text{SCN})_4]$ was synthesized and employed as the calibration standard. The synthesized metal complexes exhibited no measurable magnetic moment values. The magnetic moment of Zn(II), being a system with no unpaired electrons, confirmed its diamagnetic nature.

Thermo gravimetric study

The thermal analysis of the compounds provided insights into their stability and suggested a decomposition pathway [9]. Thermo gravimetric analysis (TGA) was conducted over a temperature range of up to 900 °C, with a heating rate of 10 °C/min and a flow rate of 20 mL/min. The thermal decomposition of the complexes occurred in four distinct steps, up to 900 °C.

The TGA data revealed that the decomposition process proceeded through several stages. Hydration water molecules were lost between 30 °C and 150 °C,

followed by the dissociation of coordinated water molecules between 150 °C and 200 °C. The decomposition of the ligand and anion commenced above 230 °C. The final stage of thermal decomposition occurred between 460 °C and 900 °C, with weight losses stabilizing at 670 °C in both complexes.

The observed weight losses closely matched the calculated values based on the proposed stoichiometry of the complexes. This indicated the complete degradation of the organic components, leaving a residue of metallic oxides in the crucible.

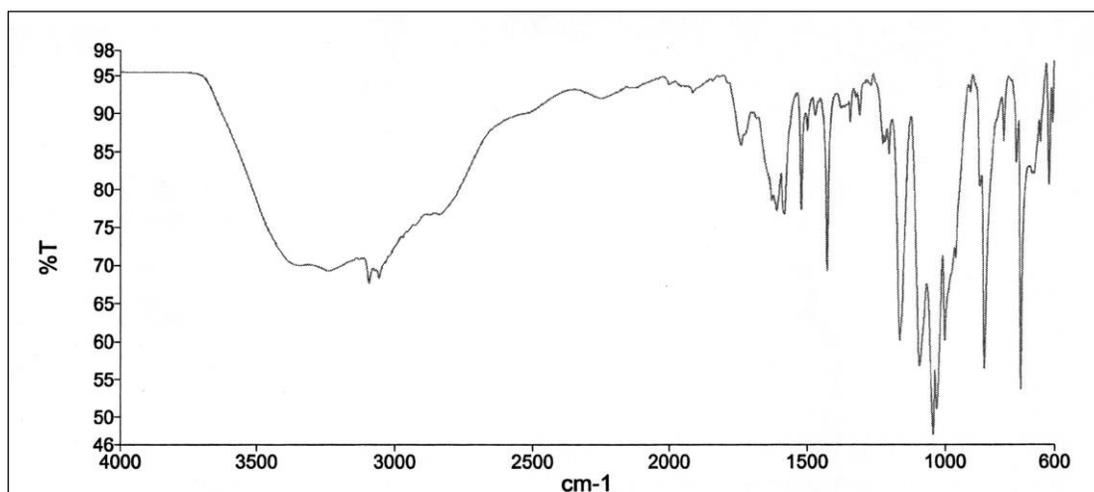


Figure 3: IR spectrum of 22

Magnetic properties

Magnetic susceptibility measurements of the

complexes were performed at room temperature using a Gouy balance. Pure $\text{Hg}[\text{Co}(\text{SCN})_4]$ was synthesized and used as

the calibration standard [11]. The synthesized metal complexes showed no measurable magnetic moment values. As

Zn(II) has no unpaired electrons, it exhibited diamagnetic behavior.

Thermo gravimetric study

Table 3: Electronic absorption bands of Ni (II) complexes and their magnetic moment values

Complexes	Electronic absorption and their assignments (nm)			Magnetic moments $\mu_{\text{eff}}\text{BM}$
	$\pi-\pi$	$n-\pi$	d-d transition	
27	290	370	615	3.17
28	285	385	620	3.15
29	280	390	690	3.22
30	282	382	665	3.20
31	286	385	655	3.18
32	282	375	630	3.14
33	288	383	673	3.16

The thermal analysis of the compounds provided insights into their stability and suggested a decomposition pathway. Thermo gravimetric analysis (TGA) was conducted up to a temperature of 900 °C, with a heating rate of 10 °C/min and a flow rate of 20 mL/min. The thermal decomposition of the complexes occurred in four distinct steps, progressing until 900 °C. The TGA data indicated that the decomposition process involved multiple

stages.

Mass spectral studies further supported the proposed constitution of the complexes. Two representative complexes, 20 and 23, exhibited molecular ion peaks at $[M^+] = 610.9890$ and $[M^{++2}] = 612.9924$, as well as $[M^+] = 642.9249$ and $[M^{++2}] = 644.9355$, corresponding to the formula weights $[\text{Zn}(\text{L})(\text{L1})\text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}$ and $[\text{Zn}(\text{L})(\text{L1})] \cdot 2\text{H}_2\text{O}$, respectively, for the nonnumeric complexes

[11]. Additionally, the spectra displayed prominent peaks corresponding to various ion fragments, providing further evidence of the molecular structure.

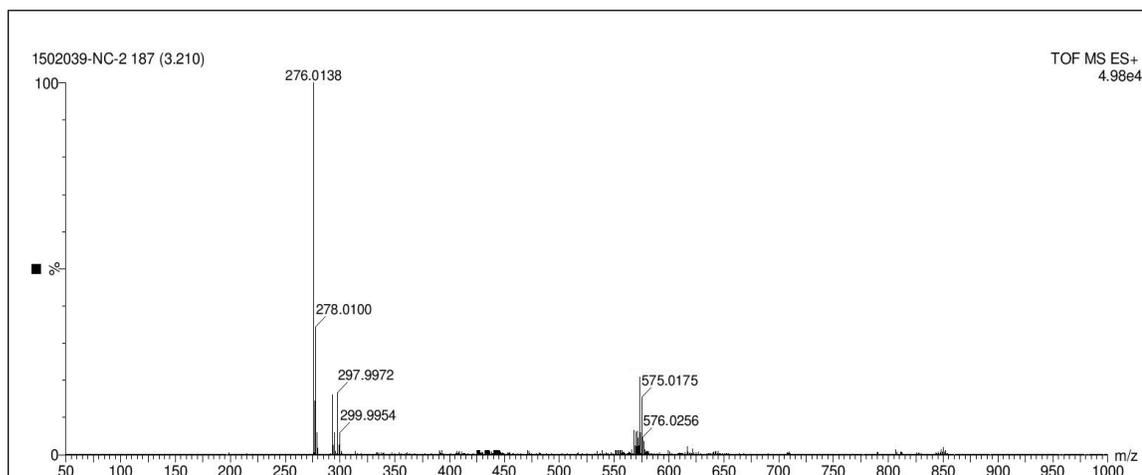


Figure 4: Mass spectrum of 14

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610.9890 and $[M^{++2}] = 612.9924$, as well as $[M^+] = 642.9249$ and $[M^{++2}] = 644.9355$, corresponding to the formula weights $[Zn(L)(L1)H_2O] \cdot 2H_2O$ and $[Zn(L)(L1)] \cdot 2H_2O$, respectively, for the monomeric complexes [11]. Additionally, the spectra displayed prominent peaks corresponding to various ion fragments, providing further evidence of the molecular structure.

Anti-oxidant Assay

Free radicals can cause DNA damage in humans, contributing to aging and various diseases, including cancer and chronic inflammation. According to the literature, certain transition metal complexes exhibit antioxidant activity. To investigate this,

experiments were conducted to assess the free radical scavenging ability of the synthesized complexes. The antioxidant potential of the complexes was evaluated using the DPPH assay, and the results indicated varying 50% inhibitory concentration (IC50) values against the DPPH radical.

The DPPH radical scavenging activity of the complexes was attributed to the presence of electron-donating atoms in their structures. In this study, the synthesized complexes demonstrated significant DPPH radical

scavenging activity, with some showing high activity based on their IC50 values. The presence of electron-releasing groups or electron-donating nitrogen groups in the ligand moiety likely contributed to the enhanced radical scavenging ability.

While certain metal complexes exhibited effective free radical scavenging properties, others showed moderate activity. These findings highlight the influence of structural features on the antioxidant potential of the complexes [13].

Table 4: Anti-fungal activity of ligands and their metal complex: Zone of inhibition in mm

Compounds	<i>sperigillus flavous</i>	<i>Fusarium</i>	<i>Phoma</i>
L1	8	8	10
14	10.2	12	17
L2	7.5	8	12
15	12.5	13.4	20.1
L3	12	6	8.1
16	14	14	19.9
L4	14	12	8
17	18.2	18.1	15
L5	16	7	12
18	17.9	16.6	17.8

L6	14	7	8
19	19	15	20
L7	2	8	15
20	5	10	10
L8	4	1	10
21	5	8	14
L9	5	7	8
22	6	10	10
L10	2	10	15
23	10	5	16
L11	2	9	10
24	5	10	12
L12	2	10	15
25	10	10	15
L1	8	8	10
26	9	12	12.8
Standard	15	20	20

The antioxidant activity of the synthesized complexes was evaluated using the DPPH assay. All the metal complexes were tested, with some demonstrating effective free radical scavenging properties, while others exhibited moderate activity [14]. This activity is attributed to the presence of electron-releasing groups or electron-

donating nitrogen groups in the ligand moiety, which significantly enhance radical scavenging potential. Complexation with metal ions further improved antioxidant activity, likely due to the presence of positively charged metal ions and the various functional groups within the moiety, which contribute to increased free radical scavenging efficiency.

Conclusion

In this study, thirteen Zn(II) complexes of N-substituted phenothiazine and 2-chlorophenothiazine ligands were synthesized and characterized. The characterization was performed using various physicochemical methods. Molar conductance measurements of all the complexes in DMF indicated their non-electrolytic nature, suggesting that the anions are coordinated to the central metal ion [15].

Magnetic and electronic spectral studies confirmed a tetrahedral geometry for all the metal complexes. Thermal analysis revealed that the complexes undergo two or three-step thermal degradation. Mass spectral analysis further validated the proposed structures and geometry of the complexes.

The antimicrobial activities of the synthesized complexes were evaluated using the disc diffusion method against three bacterial and three fungal species. The results indicated that most of the synthesized complexes exhibited notable antimicrobial activity. Additionally, the antimicrobial efficacy of the ligands significantly improved upon coordination with transition metals.

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