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Synthesis, Characterization and Investigation of Industrial, Biological Applications for Complexes Containing Diazonium Salt of 4-Aminoantipyrine

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Abstract

By reacting the diazonium salt of 4-aminoantipyrine with 2,4-dimethyphenol, 4-(2-hydroxy-3,5-dimethyl-phenylazo)- 1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one was synthesized. Elemental analysis (C.H.N.), FT-IR spectrum, UV-Vis spectroscopy, and ^1H NMR spectrum have been used to identify the ligand (L). To create complexes regarding the general formula $[M(L)_2]$ with a molar ratio (1:2), the ligand (L) and the metal ion M(II), where M(II)= (Mn, Co, and Zn), have been synthesized in an alkaline medium. UV-Vis spectroscopy, FT-IR spectra, elemental analysis (A.A.), conductivity, melting point, and magnetic susceptibility have been used to identify the complexes. These complexes exhibited a tetrahedral geometry, and their antimicrobial activity was studied using both the ligand complexes (HL) and their complexes $[M(L)_2]$. This study revealed that Mn(II), Co (II), and Zn(II) complexes have an octahedral geometry, as indicated by spectral and physicochemical data. According to activity data, the metal complexes are more effective than the free ligand. Preliminary screening has found Co(II) and Mn(II) complexes to be extremely active against all three bacterial species.

Keywords: 4-Aminoantipyrine, Azo dyes, Industrial applications, Metal complexes.

1. Introduction

Azo dyes are colored organic molecules synthesized by diazotization of a primary aromatic amine, followed by coupling agents[1,2]. Azo dyes are gaining more interest in synthetic chemistry due to their application from textile science to materials in various fields of science and technology[3-5]. Azo dyes are used in various applications, for example, in the textile industry as dyes, as paints, in the leather industry, and in the paper industry[6,7]. Recently, dyes encapsulated in polymer nanoparticles have been used for inkjet printing[8]. Polyelectrolyte membranes have also been widely used to separate dyes from wastewater by manipulating interactions between electrolytes and dyes[9]. Due to their applications of great importance, such as molecular memory storage and printing systems, which are considered among their engineering and electronic properties, complex metallic dyes have received increasing interest[10]. Coordination chemistry of transition metal complexes is a significant and enchanting branch of chemistry, with applications in industry and technology, as well as in life processes[11,12]. Without a doubt, applied sciences are quite interesting from a biomedical, chemical, and industrial standpoint. This study aims to develop and synthesize azo compounds and cobalt (II) (Co), manganese (II) (Mn), and zinc (II) (Zn) complexes from 4-(2-hydroxy-3,5-dimethyl-phenylazo)-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one, which are identified using a variety of physicochemical techniques.

2. Materials and Methods

2.1. Chemicals

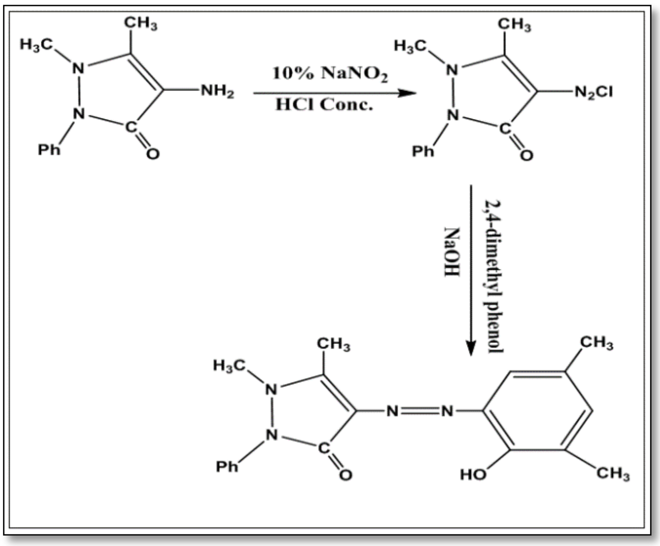
To perform the study, Fluka and Sigma-Aldrich produced all compounds used without purification.

2.2. Analytical and physical measurements

Compounds prepared from KBr pellets are measured by FT-IR using a Shimadzu FT-IR8300 Spectrophotometer in the (400-4,000) cm^{-1} range. The melting points of the compounds prepared were measured using an open capillary tube and a Stuart SMP10 melting point apparatus. Electronic spectra of every compound have been measured using a Shimadzu UV160 UV-Visible Spectrophotometer equipped with a 1 cm quartz cell, with a sample concentration of 10^{-3} M in DMSO at ambient temperature. NMR spectra using a solution of dimethyl sulfoxide (DMSO-d6 with TMS) were acquired using a 300 MHz 1H NMR spectrometer. The percentage of metals was ascertained with the use of an AA-680 Shimadzu and the atomic absorption (A.A.) technique. Conductivity measurements were carried out at room temperature and at a concentration of 10^{-3} M in DMSO using an EUTECH CON 150 conductivity meter. The magnetic moments (μ_{eff} BM) at 25 °C were measured using Sherwood Scientific magnetic susceptibility balances. The elemental analyzer EURO EA3000 was used for determining the microelemental analysis of nitrogen, carbon, and hydrogen.

2.3. Synthesis ligand (L)

It has been prepared using a modified version of the overall approach previously suggested[13]. A mixture of 10 mL of distilled water, 2 mL of 18 M hydrochloric acid, and 10 mL of ethanol has been used to dissolve 0.002 mol (0.5 g) of 4-aminoantipyrine. With constant stirring, add 10 mL of a 10% aqueous sodium nitrite solution to the solution when it has cooled between 0 and 5 degrees Celsius, being careful not to raise the temperature above that point. To complete the diazotization process and produce the diazonium salt, the solution was left for 30 minutes. After adding the salt solution incrementally while stirring constantly to 0.002 mol (0.34 g) of the 2,4-dimethylphenol alcohol solution, the solution was neutralized with sodium hydroxide to pH 5-6, after which the organic ligand precipitated. The precipitate was allowed to dry overnight before being filtered and recrystallized with ethanol, as shown in **Scheme 1**.

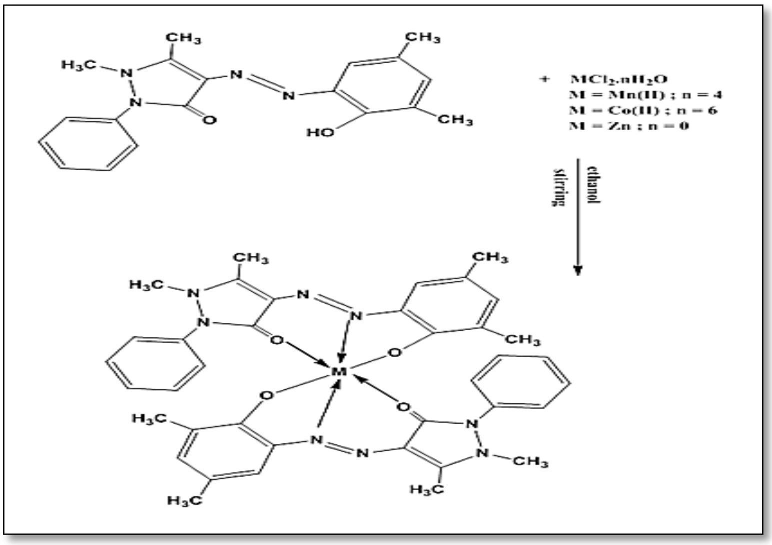


Scheme 1. Synthesis route of the ligand (L)

2.4. Synthesis of metal complexes

A drop of wisdom of stirring was added to the 0.118, 0.099, and 0.068 g from $CoCl_2.6H_2O$, $MnCl_2.4H_2O$, and $ZnCl_2$ that were resolved in aqueous solution (pH=7) using the EtOH solution from the ligand (0.336 g, 2 mmol), including 0.08 g, 2 mmol NaOH. After cooling until the precipitate turns dark, the mixture is filtered and rinsed with several ounces of a 1:1

H_2O: C_2H_5OH mixture. The preparation approach is depicted in **Scheme 2** and **Table 1** displays the physical characteristics.



Scheme 2. Proposed structure from metal (II) compounds of azo ligand (L)

Table 1. Physical properties into azo ligand also metal chelates

Compound	Colour	M.P°C	Yield%	Analysis Calc (Found)			
				M%	C%	H%	N%
Ligand(L)	Yellow	200-202	85	-	67.85 (66.88)	5.95 (5.07)	16.66 (15.92)
$[Mn(L)_2]$	Reddish brown	188 Dec	80	7.58 (6.87)	62.89 (61.92)	5.24 (4.87)	15.44 (14.76)
$[Co(L)_2$	Dark brown	266-268	83	8.09 (7.75)	62.55 (61.73)	5.21 (5.03)	15.36 (14.83)
$[Zn(L)_2]$	light brown	182 Dec	82	8.84 (7.89)	62.04 (61.81)	5.17 (4.97)	15.23 (14.72)

2.5. Microbial evaluation

The produced compounds have been estimated to assess in vitro antibacterial efficiency against Gram-positive (*Streptococcus* and *Staphylococcus aureus*) and Gram-negative (*Klebsiella* sp. and *Escherichia coli*) bacteria, as well as antifungal efficiency against *Candida albicans*, using the disk diffusion method[14]. The solvent employed for working check samples, as well as the level, has been DMSO; also, samples from (1-200 μg/mL) have been employed. Antibacterial and antifungal activities of any compound have been evaluated using the well-diffusion method. About 1 cm^3 of a 24 hr broth culture containing 106 CFU/cm^3 has been placed in sterile Petri dishes. Molten, nutritious agar (15 cm^3) has been kept at ca. 45°C and in Petri dishes, and is pliable enough to form the crucifix. Other holes with a 6 mm diameter were carefully punctured using a sterile cork borer and filled with check solutions. The dishes have been brewed for 24 hours at 37°C.

2.6. Dyeing process

Polyester, cotton, and nylon fibers are used to make and apply chemicals as dyeing grades with a 1% shade. It takes an hour to dye the fabric at 15–20°C and pH 10.

3. Results

The azo ligand (L) is suitable for diazotization with 4-bromoaniline in alkaline solution. The prepared ligand was analyzed by spectroscopic studies (^{1}H NMR, FT-IR, UV-Vis) and also trace element analysis (C.H.N.).

Solid complexes were produced by reacting the ligand dissolved in ethanol with metal ions dissolved in buffer solution (pH= 7) in a ratio of metal to ligand (1:2). Results from elemental analysis, as well as imports from metals for compounds, have been virtually identical, as were the calculated values as well. The conductivity of ligands and metal chelates dissolved in DMSO (10^{-3} mol/L) is non-electrical[15], as seen in **Table 2**.

Table 2. Electronic spectral data of compounds, and molar conductivity in DMSO (1×10^{-3} M)

Compounds	λ nm	ύ cm^{-1}	ABS	ε L. $mol^{-1}cm^{-1}$	Assignment	Λ S. $cm^2\ mol^{-1}$	μ_{eff} BM
Ligand(L)	246 382 432	40665 26178 23148	1.008 1.100 0.573	1008 1100 573	π-π^* π-π^* n-π^*	-	-
$[Mn(L)_2]$	357 445	28011 22471	1.471 0.312	1471 312	Inter ligand M.L.C.T with ${}^6A_{1g}\rightarrow {}^4Eg, {}^4T_{1g(G)}$	15.64	5.70
$[Co(L)_2]$	343 456	29154 21929	0.818 0.101	818 101	Inter ligand MLC.T with ${}^4T_{1(F)}\rightarrow {}^4T_{1(P)}$	10.72	4.80
$[Zn(L)_2]$	353 450	28328 22222	1.296 0.263	1296 263	Inter ligand Inter ligand M.L.C.T	16.21	Diamagnetic

3.1. The ¹HNMR spectrum

Figure 1 shows the ¹HNMR spectrum of the ligand (L), which displays multiple chemical shifts.

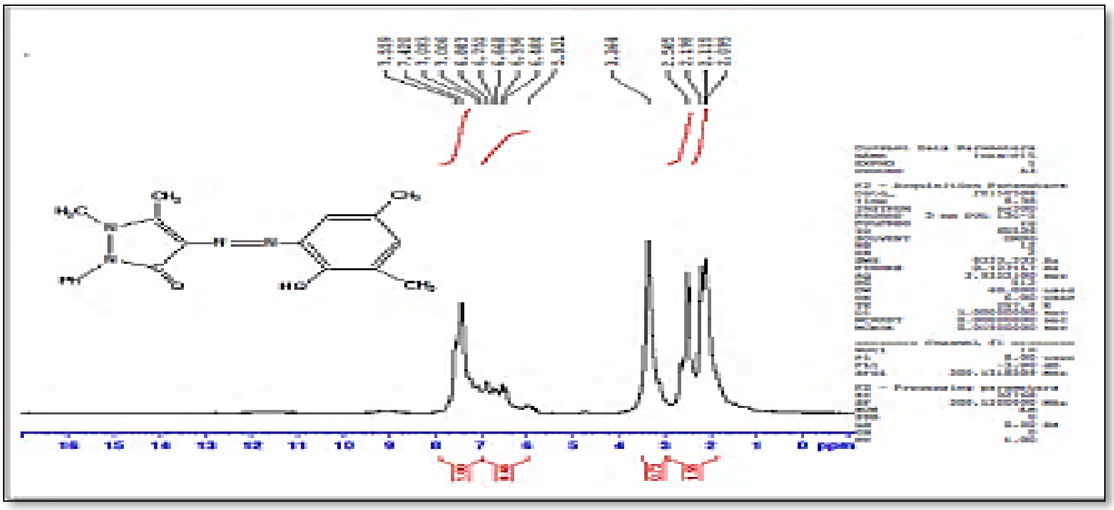


Figure 1. The ¹HNMR spectrum of ligand

3.2. Electronic spectra

UV-Vis spectra from the ligand in **Figure 2** and the prepared complexes, resolved at DMSO (10^{-3} mol/L), were measured, other than the compositional data listed in **Table 2**.

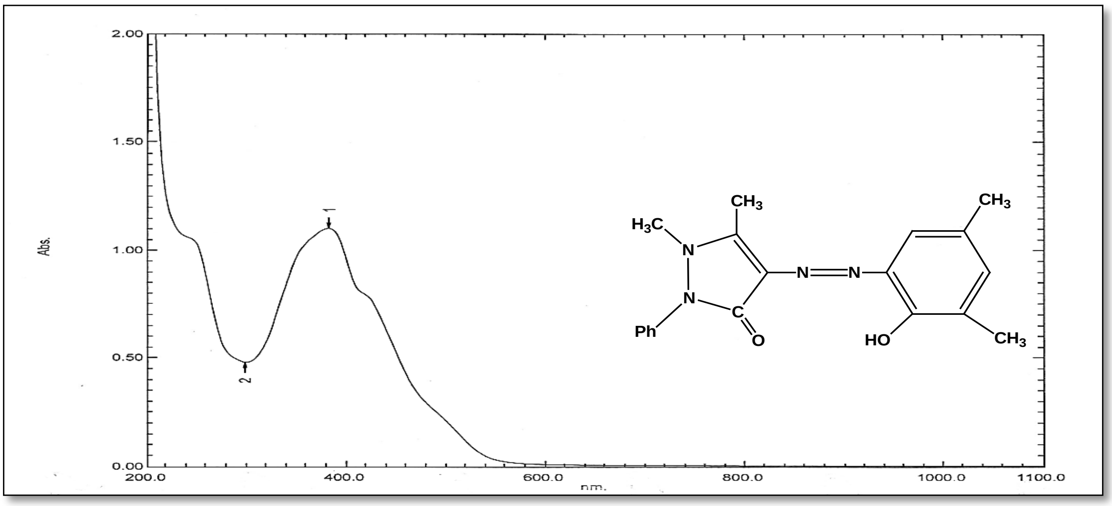


Figure 2. The UV-Vis spectrum of ligand (L)

3.3. The FTIR spectra

From summing the FTIR spectrum of the azo (L) bond, as well as metal chelates in **Figures 3-6**, with the tabulation of the data in **Table 3**.

Table 3. Infrared spectroscopic information of azo ligand (L) also its metal chelates (cm^{-1})

Compounds	**υ(OH) + υ(C=O)**	**υ(C=C) + υ(N=N)**	**δCH$_3$**	**υ(M-N) + υ(M-O)**
Ligand(L)	3411 br. 1643 sh.	1585 sho. 1527 sh.	1491 sh. 1311 sh.	-
$[Mn(L)_2]$	- 1624 sh.	1575 sh. 1491sh.	1456 sh. 1397 sh. 1311 sh.	495 w. 431 w.
$[Co(L)_2]$	- 1622 s.	1575 sh. 1490 sh.	1456 sh. 1418 s. 1397 sh. 1310 sh.	493 w. 419 w.
$[Zn(L)_2]$	- 1623 sh.	1575 sh. 1490 sh.	1456 sh. 1437 sho. 1396 sh. 1310 sh.	448 w. 428 w.

br = broad, sh = sharp, s = strong, sho = shoulder, w = weak.

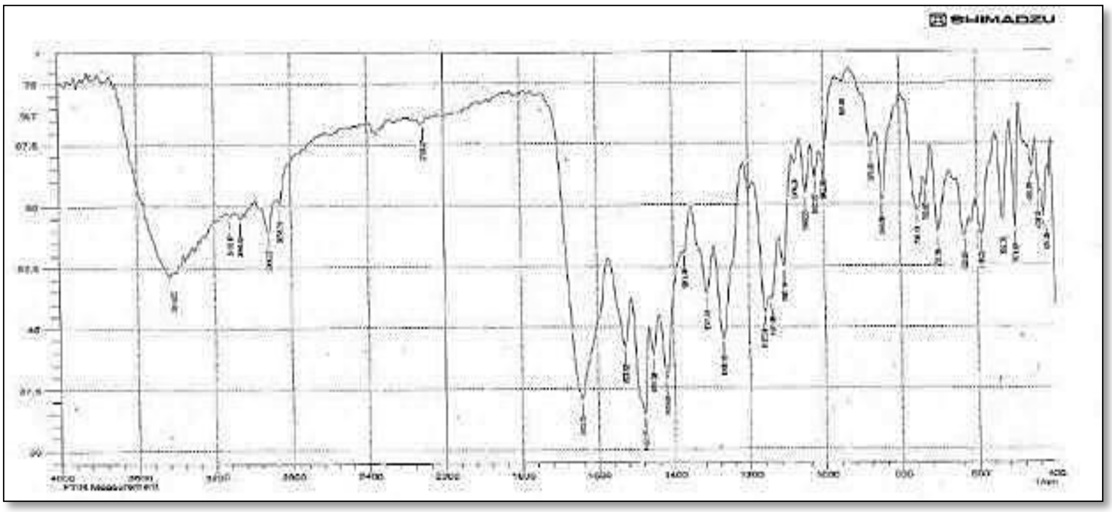


Figure 3. The FT-IR spectrum of ligand (L)

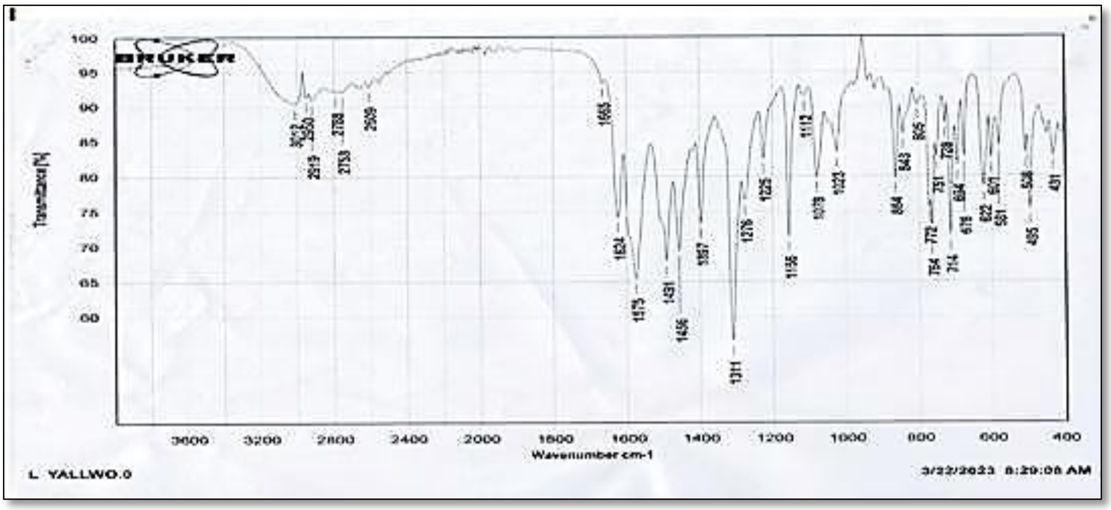


Figure 4. The FTIR spectrum of the $[Mn(L)_2]$ complex

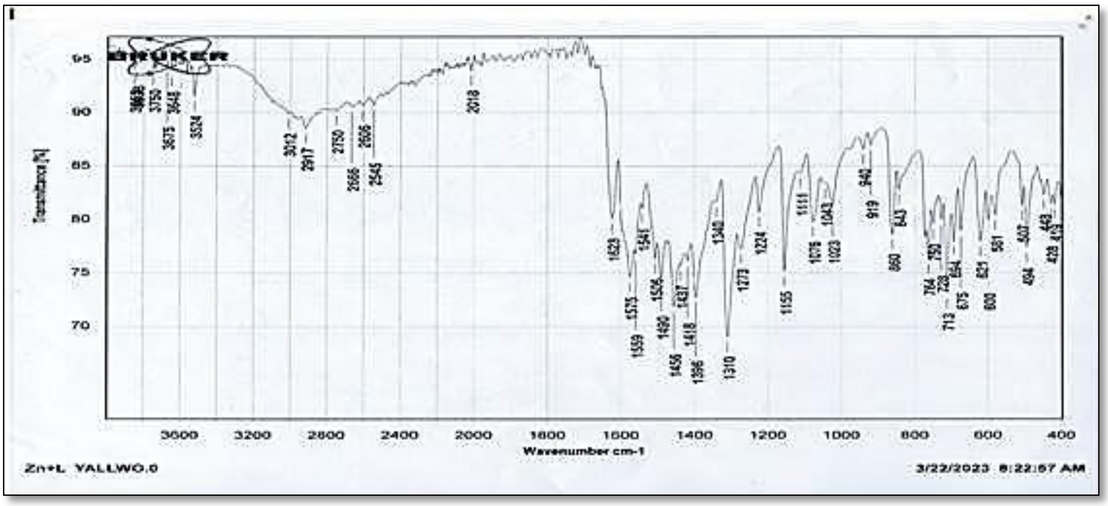


Figure 5. The FTIR spectrum of the [Co $(L)_2$] complex

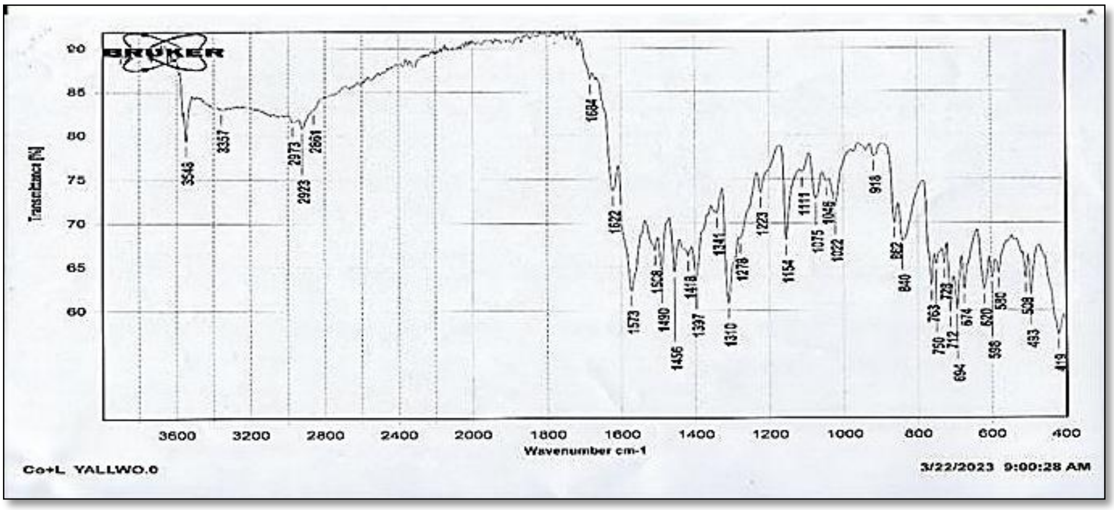


Figure 6. FTIR spectrum of the [Zn $(L)_2$] complex

3.4. Biological activity for the ligands (L) and their complexes

To assess the inhibitory effect on the growth of such organisms, the biological activity of the ligand (L) and its complexes was screened against four bacterial types: *E. coli* (a Gram-negative bacterium) and *S. aureus* (a Gram-positive bacterium). Additionally, the antifungal activity of these organisms, such as *Candida albicans*, was evaluated. The order of mixed (L) complexes' antibacterial activity was [Zn $(L)_2$] < [Co $(L)_2$] < [Mn$(L)_2$], as seen in **Table 4** and **Figure 7.**

Table 4. Inhibition circle diameter in millimeter for the ligand (L) and their complexes [M L_2]

No.	Compounds	*Staphylococcus aureus (G+)*	*Candida albicans*	*Escherichia coli (G−)*
1	(L)	12	11	12
2	[Mn$(L)_2$]	18	17	12
3	[Co$(L)_2$]	16	14	12
4	[Zn$(L)_2$]	13	14	11

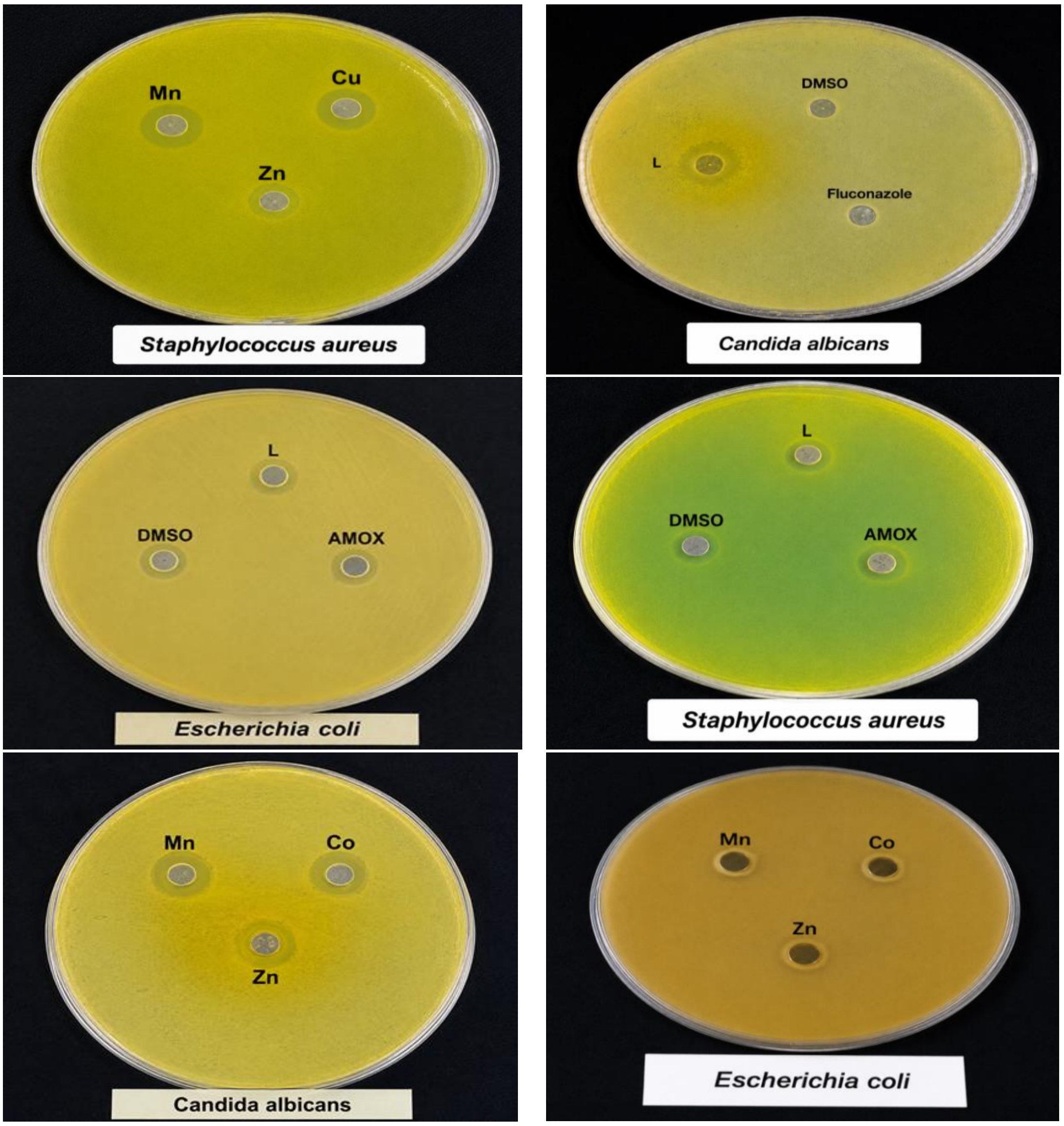


Figure 7. Biological activity for prepared compounds

3.5. Performance of dyeing

How well the finished chemicals dye cotton fabrics has already been determined. The colors affected the detergent's brightness and stability. Therefore, solid dyes have excellent color stability and texture depth. The coloring is shown in **Figure 8**.

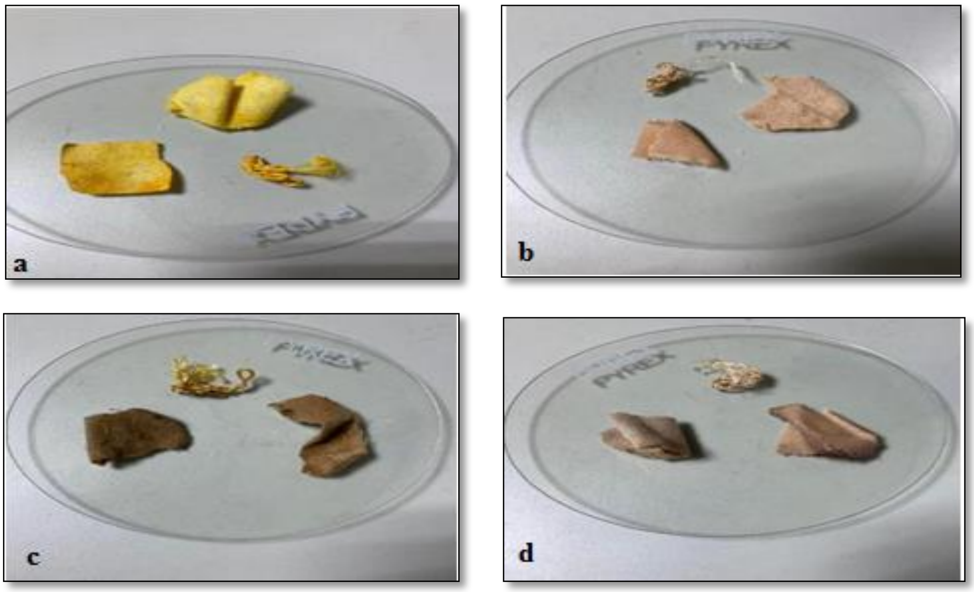


Figure 8. Textile dyeing utilizing azo ligand and metal chelates. A=L, B=L-Zn, C=L-Mn, D=L-Co

4. Discussion

4.1. The ^{1}HNMR spectra of the prepared ligands

The ^{1}HNMR spectrum of the ligand (L) showed multiple absorption bands in the range (δ = 6.484-7.559 ppm) belonging to the protons of the benzene ring[16]. The spectrum also showed an absorption band at (δ= 5.931 ppm belonging to the proton of the hydroxyl group[17], and the bands at (δ= 2.095 ppm) and (δ= 3.364 ppm) belong to $\delta(CH_3)$ and $\delta(N\text{-}CH_3)$ for the pyrazole ring, respectively[18]. As for the band at (δ= 2.198 ppm), it returns to the $\delta(CH_3)$ of the phenol ring and the band at (δ= 2.50 ppm) belongs to DMSO-d6[19].

4.2. Electronic spectra

Intense absorption peaks of the azo ligand from the spectrum width at 246 and 382 nm, which were assigned to (π-π*), as well as another peak at 432 nm due to electronic transition (n-π*)[20]. The Mn (II) spectrum shows three peaks: 357 back to intra-ligand, 445 nm, which was assigned to charge-transfer and electronic transition type $^6A_1g \rightarrow$ ^4Eg, $^4T_1g(G)$, and a magnetic moment of 5.79 B.M. This would be further evidence of octahedral geometry[21]. Co (II) complex appeared peaks on 343 nm that were appropriated into intra ligand, and another peak on 456 nm due to (M→ L) charge transfer with $^4T_1(F) \rightarrow$ $^4T_1(P)$; the magnetic moment at the compound was found on 4.80 B.M, which was closer to an octahedral environment[22]. The electronic spectrum of the Zn (II) complex shows charge-transfer bands, and the magnetic susceptibility indicates that the complex is diamagnetic because d-d transitions are not possible; hence, the electronic spectrum yielded no useful information. In fact, this result is in agreement with previous work on octahedral geometry[23].

4.3. The FTIR spectra

The extended vibration of υ (OH) of phenol revealed a broad range for the ligand at 3411 cm^{-1}. The disappearance of the ligand at this range with all complexes suggested that the phenol group was deprotonated in the coordination of the metal ion instead[24]. A strong band is observed at 1643 cm^{-1} for the pyrazole ring's υ(C=O). This band has been shifted to a lower frequency in metal chelate spectra, suggesting coordination with metal ions[25]. By lowering its frequency, the azo group's band at 1527 cm^{-1} was eliminated, indicating coordination at the metal ion[26]. The spectra of complexes gave a band at 1575 cm^{-1}, which is due to vibration from υ(C=C), and bands at 1310-1456 cm^{-1} because of the bending frequency for (δCH_3)[27]. Extending frequency bands belonging to metal-nitrogen beyond metal-oxygen, and more of them, which ensures that there are.

5. Conclusion

This study has successfully synthesized three complexes, and UV-Vis, IR, and EPR are used to characterize the 4-(2-hydroxy-3,5-dimethyl-phenylazo)-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-ligand complexes. The tridentate ligand acts. The Mn(II), Co (II), and Zn(II) complexes have an octahedral geometry, as indicated by spectral and physicochemical data. According to activity data, the metal complexes are more effective than the free ligand. Preliminary screening has found Co (II) and Mn (II) complexes to be extremely active against all three bacterial species.

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Conflict of Interest

The authors declare that they have no conflicts of interest.

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