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Metal Complexes of Cu(II) and Co(II) with 4-Aminophenol: Synthesis, Characterization and Antibacterial Test

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Abstract: The aim of this review article is to synthesize complex compounds of Cu(II) and Co(II) with 4-aminophenol and determine their formula, complex structure, characteristics, and also investigate their antibacterial activities. These complexes were synthesized by refluxing an alcoholic solution of $CuSO_4 .5H_2O$ and $CoSO_4 .7H_2O$, respectively, with 4-aminophenol in methanol for 1 hour. The products produced are characterized by using UV-Vis spectroscopy, atomic absorption spectroscopy, FTIR, thermal analysis, conductivity, and magnetic moment. The formation of the complexes were identified by shifting of the maximum wavelength toward shorter wavelength, i.e., 817 nm to 421 nm for Cu(II) complex, and 566 nm to 450 nm for Co(II) complex. Further, the proposed formulas for the complexes are $[Cu(4\text{-aminophenol})_4] SO_4$ and $[Co(4\text{-aminophenol})_4 (H_2O)_2]SO_4 .5H_2O$ with square planar and octahedral geometry respectively [1]. Both complexes are paramagnetic in nature, with slight antibacterial activity against Staphylococcus aureus, Staphylococcus epidermis, Escherichia coli, and Pseudomonas aeruginosa.

Keywords: Cobalt(II), Copper(II), Methanol, Metal complex, 4-aminophenol.

1. INTRODUCTION

4-Aminophenol or (para-aminophenol) is a derivative of phenol with an amine ($-NH_2$) group in the para position, it is an organic compound with the formula $H_2N\text{-}C_6H_4\text{-}OH$. This is commonly used as antipyretics, analgesic drugs, in photography as a developer for black and white films and marketed under the name Rodinal [2], and also used as a synthetic materials in various fields such as petroleum and rubber industry as a chemical inhibitor [3]. It is prepared from phenol by nitration followed by reduction with iron [4] Alternatively, the partial hydrogenation of nitrobenzene produce phenyl-hydroxylamine, which rearranges primarily to 4-aminophenol (Bamberger rearrangement) [5].

$$C_6H_5NO_2 + 2\ H_2 \rightarrow C_6H_5NHOH + H_2O$$
$$C_6H_5\text{-}NH\text{-}OH \rightarrow HO\text{-}C_6H_4\text{-}NH_2$$

The 4-aminophenol has electron donor amine ($-NH_2$) group and hydroxy (-OH) group. While forming complex compounds the 4-aminophenol can be used as ligand, as it can donate electron pairs to the metal ion so as to form the complex compound.

Metal complexes are the complex compounds consisting of a central metal atom or ion surrounded by number of neutral molecules or negatively charged ions called ligands. Which can donate number of pair of electrons to the central metal atom or ion to form coordinate covalent bonds with the central metal atom or ion. These compounds are stable and retain their properties in the solid state as well as in their solution [6]. Metal complexes have variety of applications in the field of analytical chemistry, biological science, pharmacy, medicine, clinics, industry, and others. In addition, most of the transition metal complexes and ligands have been widely used as drugs, edta4- is used to remove excess of lead as lead complex, chelating ligands like D-penicillamin and desferrioxime β are used to remove excess copper, a complex of platinum $[Pt(NH_3)_2 Cl_2]$ is used to inhibit the growth of tumors in the body. In designing effective complex compounds, the choice of metal ion structure and ligand is an important factor [7]. Transition metal complexes containing copper, cobalt, nickel, zinc, and silver, along with ligands, are commonly employed in pharmaceutical

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applications for their antibacterial, anticancer, and antioxidant properties [7,8]. Copper is widely used in the synthesis of complex compounds which are widely used in medicine, because it is proven that Cu(II)-metal complexes have high antibacterial power and low toxicity, so Cu(II) ions are used as an antibacterial substitute for silver [9]. The another transition metal atom that has been widely used is Co(II). The complex compound which were prepared by Mishra et al. [10], the N and 'O' groups of the ligands bind to metal ions Co(II), Cu(II), Ni(II), Zn(II), and Cd(II). Herrera et al. [11] synthesized a complex of Co(II), Ni(II), and Cr(III) with 2-thiozoline-2-thiol and showed better antibacterial activity. Al-Zaidi et al. [11] A Co(II) complex was synthesized using a ligand called 1-(4-(4(diethylamino)-2-hydroxybenzylidene amino) phenyl, ethanone oxime. The findings revealed that the complex exhibited enhanced antibacterial properties when compared to both the ligand and its complex ion. In a separate study, Prajapati et al. [13] synthesized Co(II) with 2-[(E)-(2-hydroxybenzylidene)amino] benzoic acid and observed favorable antibacterial activity. On the bases of the above clarification, 4-aminophenol has the capability to be a ligand which can coordinating with Cu(II) and Co(II). Based on our knowledge, both complexes with identical structure never been synthesized before. Hereby, the $[Cu(4\text{-aminophenol})_4]SO_4.H_2O$ and $[Co(4\text{-aminophenol})_4(H_2O)_2]SO_4$ $.5H_2O$ complexes have been effectively synthesized and characterized. The complexes were also tested for antibacterial properties against Staphylococcus aureus, Staphylococcus epidermis, Escherichia coli, and Pseudomonas aeruginosa.

2. INSTRUMENTATION

The molar absorption and electronic transition of the solutions of both metal complexes were estimated by double-beam UV-Vis spectroscopy (Shimadzu PC 1601). The formula of the complexes were determined by determining the metal ion concentration of the complexes using an atomic absorption spectrophotometer (AAS, Shimadzu AA-665). The presence or absence of water molecules in the complex were determined using a thermal gravimetric analysis/differential scanning calorimetry instrument (TG/DSC, Shimadzu 50) with a heating rate of 10 °C/min. Electrical conductivities of Cu(II) and Co(II) complexes and solutions of some known metal salts in DMF and DMSO, respectively, at 25 °C using a conductivity meter (Jenway CE 4071). Infrared spectra of 4-aminophenol and the complex were measured using a Fourier-transform infrared spectrophotometer (FTIR, Prestige-21 Shimadzu) in the frequency range of 4000–450 cm^{-1} as KBr pellets. The magnetic properties of powdered Cu(II) and Co(II) complexes can be moment determined with a magnetic susceptibility balance (MSB, Auto Sherwood Scientific 10169) and corrected using Pascal's constant.

3. METHODOLOGY

3.1. Chemicals used: $CoSO_4$ $.7H_2O$, $CuSO_4$ $.5H_2O$, $CuCl_2$ $.2H_2O$, $CoCl_2$ $.6H_2O$, 4-aminophenol, Methanol.

3.2. Synthesis of Co(II))-4-aminophenol complex

The Co(II))-4-aminophenol complex, was synthesized by dissolving 0.436 gm. of 4-aminophenol in 10 ml. methanol, and 0.281 gm of metal compound CoSO 4 $.7H_2O$ was also dissolved in 10 ml. methanol separately. The ligand solution (4-aminophenol) was heated and stirred constantly with a magnetic stirrer and then it is added drop wise into the metal solution. The mixture was refluxed for 1 hour at 60 °C. The solution is then concentrated until its volume reduces to half of the initial volume, then it is allowed to stand for crystallization. The crystals formed were filtered, washed with acetone and dried under vacuum.

3.3. Synthesis of Cu(II)-4-aminophenol complex

The Cu(II)-4-aminophenol complex was synthesized in a ratio of 1 : 4 by dissolving 0.249 gm of metal compound $CuSO_4$ $.5H_2O$ in 10 ml. methanol, and 0.436 gm. of 4-aminophenol in 10 ml. methanol separately. The 4-aminophenol ligand solution was heated on a hot plate with constant stirring using amagnetic stirrer, and then it is added drop wise into the metal solution. The mixture was then refluxed for one hour, at 67 °C. The solution is then concentrated until its volume reduces to half of the initial volume, then it is allowed to stand for crystallization. The crystals formed were filtered, washed with acetone and dried under vacuum.

3.4. Antibacterial test of Cu(II)- 4- aminophenol, and Co(II)- 4- aminophenol complexes

Antibacterial activity of Cu(II)- 4- aminophenol, and Co(II)- 4- aminophenol complexes against S. aureus ATCC 25923, S. epidermis ATCC 12228, E. coli ATCC 25922, and P. aeruginosa ATCC 27853 was tested using paper disc diffusion method. The inhibition of bacteria was shown as the diameter of the clear zone in the test samples. The three test samples, namely metal compound, $CuSO_4$ $.5H_2O$, $CoSO_4$ .7H 2 O and the ligand 4- aminophenol. Cu(II)-4-

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aminophenol and Co(II)-4-aminophenol complexes were made with various concentrations in ppm. as, 125, 250, 500, and 1000 ppm.

4. INDICATION OF THE FORMATION OF METAL COMPLEXES

When the alcoholic ligand solution of 4-aminophenol is added to the alcoholic solution of $CoSO_4 .7H_2O$, the change in color of complex solution to blackish brown indicates the formation of cobalt(II)- 4- aminophenol complex. The shift in the absorption of the, maximum of wavelength from 566 nm., towards a smaller wavelength of 450 nm. can be seen in Fig. 1. The shift of the maximum wavelength indicates the formation of complexes between Co(II) and 4-aminophenol ligands [14]. Similarly, Co(II)- nicotinamide complex that was synthesized by Rahardjo et al. [15] also experienced a shift towards a lower wavelength from 512 nm,. to 506 nm., (complex solution).

Meanwhile, the formation of the copper(II)-4aminophenol complex was indicated by change in color from brown to black. The synthesis product was black precipitates (0.396 g; 64.39%) [14] Fig. 2 shows a shift in the maximum wavelength absorption (λ max) of $CuSO_4 .5H_2O$ (817 nm) towards a lower wavelength in the Cu(II)-4 aminophenol complex (421 nm). The shift in wavelength indicates the formation of Cu(II)-4- aminophenol complex. It also proves that 4-aminophenol has a stronger ligand field energy than H_2O so that it is able to substitute H_2O positions. Other syntheses of copper(II) complexes also experienced a maximum wavelength shift, from 700 nm to 380 nm. [16].

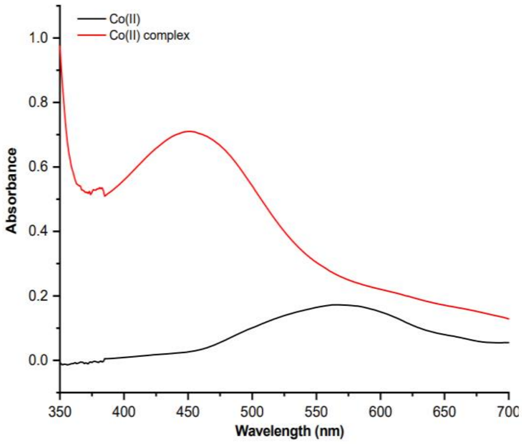

Fig 1. UV-vis spectra of Co(II) and its complex in methanol

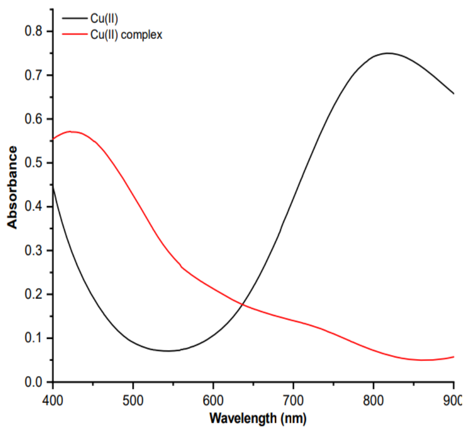

Fig 2. UV-vis spectra of Cu(II) and its complex in methanol

4.1. Atomic Absorption Spectrophotometer (AAS)

The results of the measurement of copper and cobalt content experimentally with AAS in Cu(II)-4-aminophenol were 10.59 • } 0.22% and 8.62 • } 0.20%, respectively (Table 1). The results of the measurement of copper and cobalt content are compared with theoretical Cu levels in various possible
complex formulas as shown in Table 1, it can be estimated that the complex compound formula Cu(II)-(4-aminophenol) is Cu[(4- aminophenol) $4SO_4$ $(H_2O)n$] [14]. (n = 0, 1, or 2) and Co[(4-aminophenol)$_4SO_4$ $(H_2O)n$]. (n = 5, 6, or 7).

4.2. Thermal Analysis

The thermogram of the Cu(II)-4-aminophenol complex in Fig. 3 indicates the occurrence of a mass shift of the complex to the extent that 3.12% at a temperature of 30–155 °C equivalent to the release of one H_2O molecule (theoretical calculation: 2.93%). Therefore, the empirical formula of Cu(II) complex is Cu[(4- aminophenol) $4(SO_4)(H_2O)$]. For Co(II) complex, the thermogram shows a double decrease in mass (Fig. 4). The first decrease occurred at a temperature of 40–135 °C and the second decrease occurred at a temperature of 135–185 °C. At a temperature of 40–135 °C there is a decrease in mass of 13.90% which is equivalent to five H_2O molecules (theoretical calculation: 13.205%). The second decrease was reduced by 6.06% equivalent to the release of two H_2O molecules (theoretical calculation: 5.74%). According to Himawati et al. [17], the reduce in a temperature of 30–130 °C water molecules as hydrated water molecule but not as a ligand, whereas according to Prajapti et al. [13], reducing mass at a temperature of 150–250 °C is water coordinated with metals. Co-[4- aminophenol]$_4(SO_4)(H_2O)_7$. is the empirical formula for the Co(II) complex

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with 4-aminophenol. Empirical formula of the Co(II)- complex with 4-aminophenol is Co[(4- aminophenol). 4(SO_4)(H_2O)$_7$]

4.3. Molar Conductivity

The molar conductivity of the standard solution in Table 2 shows that the more ions produced in the solution, the greater the value of molar conductivity.

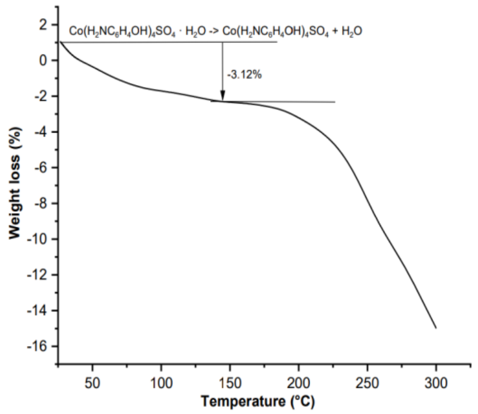

Fig 3. Thermogram of Cu(II) complex

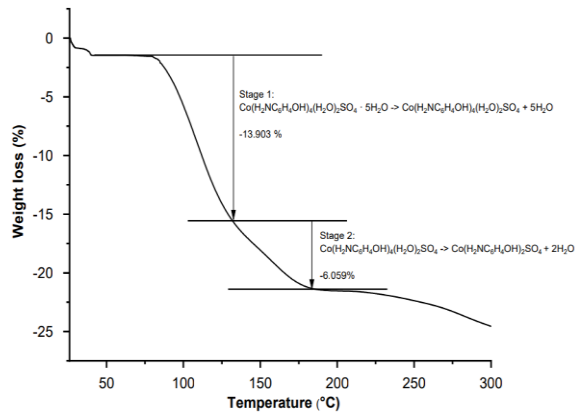

Fig 4. Thermogram of Co(II) complex

Table 1. Proposed formula of the complex based on AAS result

Proposed Empirical Formula	Theoretical metal content (% of Cu or % of Co)	AAS Result (%)
Cu-(4-aminophenol)$_4$$SO_4$	10.73	
Cu-(4-aminophenol)$_4$(SO_4)(H_2O)	10.41	10.59
Cu-(4-aminophenol)$_4$(SO_4)(H_2O)$_2$	10.11	
Co-(4-aminophenol)$_4$(SO_4)(H_2O)$_5$	8.66	
Co-(4-aminophenol)$_4$(SO_4)(H_2O)$_6$	8.42	8.62
Co-(4-aminophenol)$_4$(SO_4)(H_2O)$_7$	8.22	

Table 2. Average molar conductivity of the complexes and metal salts

Solvent	Compounds	Average molar conductivity (Scm2/mol)	Number of ions	Average molar conductivity of the complex
	$CoSO_4.7H_2O$	10	2	
DMF	$CuSO_4.5H_2O$	16	2	[Cu (4-aminophenol)$_4$] $SO_4.H_2O$ = 16 S cm^2/mol
	$CuCl_2.2H_2O$	28	3	
	$FeSO_4.7H_2O$	6	2	
DMSO	$CoSO_4.7H_2O$	10	2	[Co(4-aminophenol)$_4$(H_2O)$_2$] SO_4 .5H_2O = 10 S cm2/mol
	$CuCl_2.2H_2O$	32	2	
	$CoCl_2.6H_2O$	86	3	

The molar conductivity of the complex sample solution can be compared to the molar conductivity of the standard solution. This comparison reveals the number of ions produced in the sample solution. The molar conductivity of

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Cu(II)-4-aminophenol complex solution in DMF calculated by electrical conductivity is close up to the value of the electrical conductivity of the $CuSO_4$.$5H_2O$ solution, which indicate the number of ions in the complex is 2. This indicates that the complex solution is an electrolyte with a ratio of cation and anion charges. in a 1:1 ratio. Thus, the sulfate in the complex does not act as a ligand but as a counter ion. Therefore, the possible complex formula for Cu(II)-4-aminophenol is [Cu(4- aminophenol)$_4$]SO_4 .H_2O.

The molar conductivity of the Co(II)-4- aminophenol complex solution in DMSO is similar to the value of the molar conductivity of the $CoSO_4$.$7H_2O$ solution, which both have a value of 10. This indicates that the number of ions contained by the Co(II)-4- aminophenol complex is two ions. This shows that the ratio of the total charge of cations and anions in the Co(II)-4-aminophenol complex is 1:1, so the formula for the Co(II)-4-aminophenol complex is [Co-(4-aminophenol)$_4$(H_2O)$_2$]SO_4 .$5H_2O$ [14], with $SO_4{}^{2-}$ as the counter ion.

4.4. FTIR

FTIR absorption can be used to estimate the functional groups that attach to the central metal ion. The absorption range for -OH phenol is 3531 cm^{-1} to 3640 cm^{-1}, while H_2O has a range of 3200 cm^{-1} to 3570 cm^{-1} (broad) [18]. A shift in C-N absorption from 1612 cm^{-1} to 1607 cm^{-1}, as shown in Fig. 5,
suggests the formation of a Cu(II) complex compound [19]. Similar shifts in absorption were observed in the Cu(II) complex with 1,3,4-thiadiazolethiosemicarbazone, with -NH_2 absorption shifting from 3264 cm^{-1} to 3246 cm^{-1} and from 3152 cm^{-1} to 3134 cm^{-1}, and C-N absorption shifting from 1605 cm^{-1} to 1599 cm^{-1} [20]. The coordination of the nitrogen atom to copper was also visible in the Cu(II) complex with Schiff base-on glycine, where C-N absorption changed from 1641 cm 1 to 1600 cm $^{-1}$. [21]. In this study, the -NH_2 absorption in the ligand and [Cu(4-aminophenol)$_4$]SO_4 shifted to smaller values of 3352 cm^{-1} and 3287 cm^{-1}, respectively [14] This suggests that the -NH_2 group is coordinated through the nitrogen atom to 4-aminophenol on the Cu(II). The hydroxyl (-OH) group is not visible in the spectra, probably because it overlaps with -NH_2 group. The presence of Cu-N absorption at 417 cm^{-1} supports this. Batool et al, [22], also noted that a new absorption at 414 cm^{-1} indicates the presence of metal bonds with the -NH_2 (Cu-N).

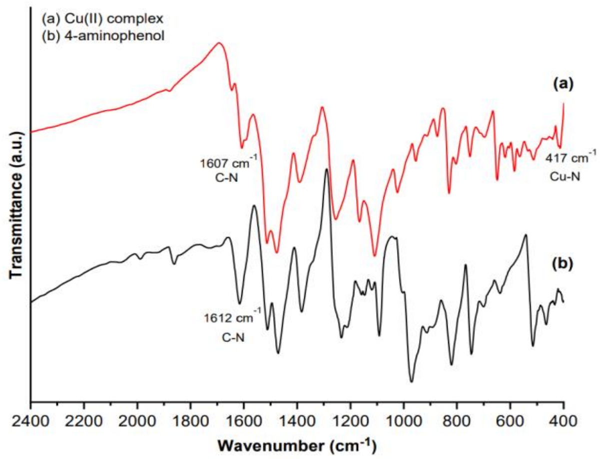

Fig 5. FTIR spectra of 4-aminophenol and Cu(II) complex

In Co(II) complex, NH_2 and C–N absorption shifted toward smaller from 3342 (4-aminophenol) to 3282 cm^{-1} (complex) and 1385 to 1384 cm−1 (Fig. 6). A similar case also happened to [Co(Phen)$_2$(H_2O)$_2$] Cl_2 .H_2O, C–N absorption shift from 328 to 1314 cm^{-1} [23]. Absorption of water molecules does not appear. However, the presence of absorption at 524 cm^{-1} indicates the presence of Co–N bonds. This shows that in the complex [Co(4-aminophenol)$_4$(H_2O)$_2$]SO_4.$5H_2O$, the cobalt ion binds to the nitrogen atom of -NH_2 and the oxygen atom of H_2O.

4.5. UV-Vis

[Cu(4-aminophenol)$_4$]SO_4 .H_2O showed an absorption at 417 nm (23981 cm$_{-1}$), as revealed in Fig. 1. Copper complexes that have electronic absorption in the region of 20366 and 20202 cm^{-1} have 2B1g→2E1g transitions and have a square planar geometry [24]. Thus the complex of [Cu(4 aminophenol)$_4$]SO_4].H_2O is estimated to have square planar geometry. The Co(II) complex, [Co(4- aminophenol)$_4$ (H_2O)$_2$]SO_4 .$5H_2O$ showed one absorption peak at a wavelength of 450 nm (22,222 cm^{-1}). Co(II) complex with Schiff base salicylidenic ligand at a maximum wavelength of 470 nm

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(21,276 cm^{-1}) has an electronic transition of 4T1g(F) → 4T1g(P) and has octahedral geometry [25]. Thus, [Co(4-aminophenol)$_4$ $(H_2O)_2$]SO_4 .5H_2O is also estimated to have octahedral geometry. Electronic spectral data of [Cu(4-aminophenol)$_4$]SO_4 .H_2O and [Co(4- aminophenol)$_4$ $(H_2O)_2$]SO_4 .5H_2O are shown in Table 3.

4.6. Magnetism

The results of the measurement of the effective magnetic moment (μ_{eff}) of [Cu(4- aminophenol)$_4$]SO_4 .H_2O and [Co(4-aminophenol)$_4$ $(H_2O)_2$]SO_4 .5H_2O is 1.79 and 5.30 BM, respectively. [Cu(4-aminophenol)$_4$]SO_4 .H_2O has similar magnetic properties to other Cu(II) complexes in the range 1.70–2.0 BM, which show paramagnetic characteristics with one unpaired electron [19,26,27,28]. The result of [Co(4-aminophenol)$_4$ $(H_2O)_2$]SO_4 .5H_2O is similar to the effective magnetic moment value of the copper complex according to [Co(Nicotinamide)$_2$ $(H_2O)_2$]Cl_2 , which was reported by Rahardjo et al. [15] was 5.3 BM and Obaid et al. [29] was 5.25. The high spin octahedral Co(II) geometry has an effective magnetic moment value of 4.7–5.3 BM [23]. The significantly higher moment magnetic of octahedral Co(II) complexes than that of spin-only value (μs, 3.87 BM) for three unpaired electrons is due to the orbital contribution to the magnetic moment [31-32]. The proposed structures of the Cu(II) and Co(II) complexes are shown in Fig. 7.

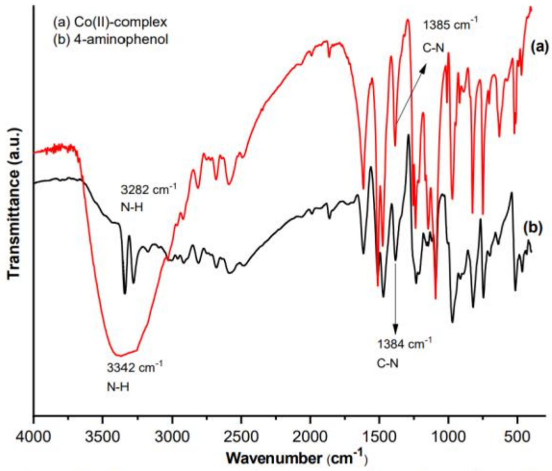

Fig 6. FTIR spectra of 4-aminophenol and Co(II) complex

Table 3. Electronic spectral data of [Cu(4-aminophenol)$_4$]SO_4.H_2O and [Co(4-aminophenol)$_4$ $(H_2O)_2$]SO_4.5H_2O

Compounds	λ_{max}	Absorbance	ν (cm−1)	ε (L mol−1 cm−1)
$CuSO_4$・5H_2O	794	0.146	12595	91.074
$CoSO_4$・7H_2O	566	0.17	12592	35
[Cu(4-aminophenol)$_4$]SO_4.H_2O	417	0.777	23981	795.35
[Co(4-aminophenol)$_4$$(H_2O)_2$]$SO_4$.5$H_2O$	450	0.71	22222	830

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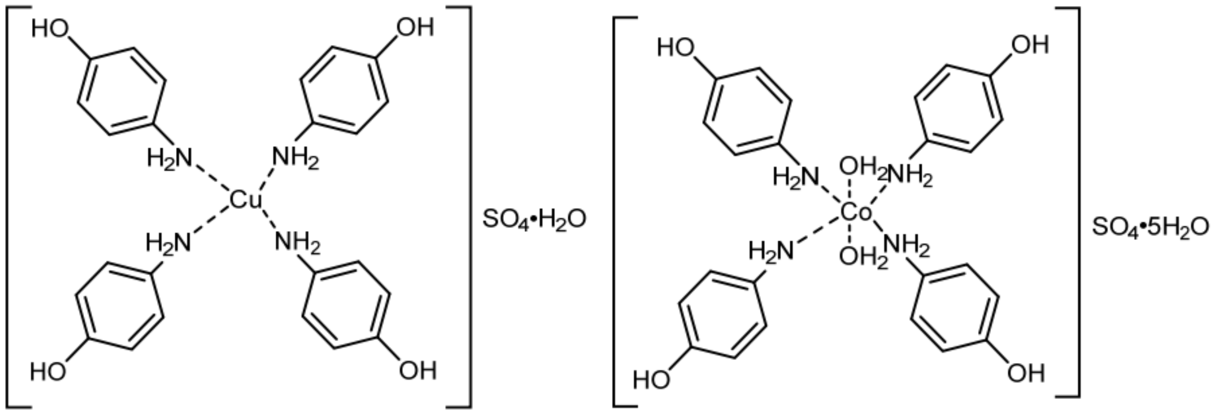

Fig 7. Proposed structures of Cu(II) and Co(II) complexes

4.7. Antibacterial Test

Both [Cu(4-aminophenol)$_4$]SO_4 .H_2O and [Co(4-aminophenol)$_4$ (H_2O) $_2$]SO_4 .5H_2O do not exhibit antibacterial properties against S. aureus ATCC 25923, S. epidermis ATCC 12228, E. coli ATCC 25922, and P. aeruginosa ATCC 27853.

5. CONCLUSION

Copper and cobalt compounds have been effectively produced with estimated formulas [Cu(4-aminophenol)$_4$]SO_4 .H_2O and [Co(4-aminophenol)$_4$ (H_2O)$_2$]SO_4 .5H_2O. These complexes exhibit paramagnetic properties and are believed to adopt square planar and octahedral geometries, respectively. However, no antibacterial effects were observed against S. aureus, S. epidermis, E. coli, and P. aeruginosa.

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