Composition, Characterization and Antibacterial activity of Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) mixed ligand complexes Schiff base derived from Trimethoprim with 8-Hydroxy quinoline

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Composition, Characterization and Antibacterial activity of Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) mixed ligand complexes Schiff base derived from Trimethoprim with 8-Hydroxy quinoline

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Abstract. New Schiff base ligand 2-((4-amino-5(3, 4, 5-trimethoxybenzyl) pyrimidin-2-ylimino) (phenyl)methyl)benzoic acid = [HL] was synthesized using microwave irradiation trimethoprim and 2-benzoyl benzoic acid. Mixed ligand complexes of Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) are reacted in ethanol with Schiff base ligand [HL] and 8-hydroxyquinoline [HQ] then reacted with metal salts in ethanol as a solvent in (1:1:1) ratio. The ligand [HL] is characterized by FTIR, UV-Vis, melting point, elemental microanalysis (C.H.N), 1H-NMR, 13C-NMR, and mass spectra. The mixed ligand complexes are characterized by infrared spectra, electronic spectra, (C.H.N), melting point, atomic absorption, molar conductance and magnetic moment measurements. These measurements indicate that the ligand [HL] coordinates with metal (II) ion in a tridentate manner through the oxygen and nitrogen atoms of the ligand, octahedral structures are suggested for these complexes. Antibacterial activity of the ligands [HL], [HQ] and their complexes are studied against (gram positive) and (gram negative) bacteria.

1. Introduction

Schiff bases have played a significant role in the development of coordination chemistry and have been implicated as an important point in the development of inorganic biochemistry and optical materials [1]. Schiff base metal complexes have been widely studied because they have industrial, antifungal and biological applications [2-4]. Schiff bases have played a significant role in the development of coordination chemistry and have been implicated as antreatment and urinary tract infections [5]. Antibiotic 5-(3, 4, 5-trimethoxybenzyl) pyrimidine-2, 4-diamine (Trimethoprim) is used mainly in the treatment of urinary tract infections [6]. Chelating ligands containing O and N donor atoms show broad biological activity and are of special interest because of the variety of ways in which they are bonded to metal ions [7]. Mixed ligands complexes have been studied because of their importance in biological systems [8-9, 10]. Few reports are available on the mixed metal complexes of trimethoprim [11]. Thus, this work is aimed at the synthesis, characterization and biological studies of mixed ligand complexes of trimethoprim and 2- benzoyl benzoic acid Schiff base and mixed ligand complexes with 8-hydroxy quinoline .The metal ion in an octahedral environment. The primary aim of the current study to
determine the structure and geometry of tridentate Schiff base mixed ligand Mn (II), Co (II), Ni (II), Cu (II), Zn (II) and Cd (II) metal complexes.

2. Experimental

2.1. Materials and Methods
All Chemicals employed were of analytical grade and used without further purification. The reagents were used without further purification. FTIR spectra were measured as (KBr disc) utilizing "Shimadzu FTIR-8400S", Fourier Transform Infrared spectrophotometer. ¹HNMR and ¹³CNMR were carried out by using Bruker500 MHz NMR spectrophotometer in central laboratory Isfahan University. Mass spectra were obtained by using Acq Method LOW ENERGY DSD Direct Probe, central lab Isfahan University, Iran. The electronic spectra were recorded in DMSO on a "Shimadzu UV-visible-160 A Ultra Violet-Visible Spectrophotometer". Elemental microanalyses (C.H.N.) were performed by using a Leco 932 USA Elemental Analyzer. The atomic absorption was measured by using "Varian-AA 775 Atomic Absorption spectrophotometer". Conductivity measurements were made in DMSO for 10⁻³ M of complexes by using (Philips PW9526 Digital Conductivity meter) at room temperature. Magnetic moment (µ eff B.M) for the prepared complexes was measured at room temperature by using Bruker Magnet B.M. Finally, melting points were got by utilizing "Stuart Melting Point Apparatus".

2.2. Synthesis of ligand Schiff Base
(0.1g, 0.34 mmole) trimethoprim is mixed with (0.09g, 0.34 mmole) 2-benzoylbenzoic acid in (1:1) ratio mole, and the mixture is grinded in ceramic mortar. Then, the contents are subjected to microwave irradiation at (100 Co) for 10 minutes. The reaction product is washed with small portion of benzene as off white of ligand. Weight (0.16g), yield (94%), m.p (96-98)°C. The ligand [HL] is recognized by FTIR spectral, ¹H and ¹³C NMR, mass spectrum and elemental analysis. The synthesis route of the ligand is shown in figure(1).The microanalysis of results for the ligand [HL] and some of its physical properties are given in Table 1.

2.3. General synthesis of mixed ligands complexes
An ethanolic solution (25ml) of metal salts of Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) was added gradually to a stirred ethanolic solution of Schiff base(25ml) and secondary ligand 8-hydroxyquinoline ethanol to get (1:1:1) (metal:HL:HQ) molar ratio. Few drops of KOH solution were added to adjust the pH < 9. The resulting solution was refluxed for about 2 hours. The chelate was precipitated, cooled and then filtered. The result got by washed with small amounts of ethanol and dried. The microanalysis of results for the ligand and some of its physical properties are given in Table 1.
Figure 2. Suggested structure for mixed ligand complexes

Table 1. Some of physical properties and microanalysis of all prepared product

| Compound         | Empirical Formula (formula wt.) | M.P Dec. | Yield % | Color   | Elemental Analyses Found (Calc.) % | Molar conductivity (ohm cm⁻¹ mol⁻¹) |
|------------------|---------------------------------|----------|---------|---------|----------------------------------|-----------------------------------|
|                  |                                 |          |         |         | C          | H          | N          | M          | Cl          |                                |
| [HL]             | C₂₂H₂₈N₄O₁₅                   | 498.35   | (96-98) | Off white | 66.34 (66.46) | 5.53 (4.26) | 10.19 (10.24) | -           | -           |                                |
| [Mn(L)(Q)(H₂O)]  | C₃₇H₅₃MnN₇O₁₁               | 714.63   | 300 < dec. | Brown   | 62.19 (61.25) | 4.65 (4.39) | 9.80 (8.74) | 7.69 (7.48) | Nil 12.3                |                                |
| [Co(L)(Q)(H₂O)]  | C₃₇H₅₃CoN₇O₁₁              | 718.62   | 300 < dec. | Olive   | 61.84 (60.65) | 4.63 (3.39) | 9.75 (9.24) | 8.20 (7.88) | Nil 9.9                 |                                |
| [Ni(L)(Q)(H₂O)]  | C₃₇H₅₃NiN₇O₁₁              | 718.38   | 300 < dec. | Pale green | 61.86 (61.67) | 4.63 (4.39) | 9.75 (8.25) | 8.17 (7.95) | Nil 8.7                 |                                |
| [Cu(L)(Q)(H₂O)]  | C₃₇H₅₃CuN₇O₁₁              | 723.23   | 300 < dec. | Green   | 61.45 (60.89) | 4.60 (3.36) | 9.68 (8.99) | 8.79 (8.34) | Nil 10.6                |                                |
| [Zn(L)(Q)(H₂O)]  | C₃₇H₅₃ZnN₇O₁₁              | 725.10   | 300 < dec. | Yellow | 61.29 (58.15) | 4.59 (4.35) | 9.66 (9.16) | 9.02 (8.96) | Nil 8.5                 |                                |
| [Cd(L)(Q)(H₂O)]  | C₃₇H₅₃CdN₇O₁₁              | 772.10   | 300 < dec. | Yellow | 57.56 (56.78) | 4.31 (4.10) | 9.07 (8.73) | 14.56 (13.88) | Nil 10.2                |                                |

dec. = Decomposition

3. Results and Discussion
Spectroscopic [FT-IR, ¹H, ¹³C-NMR,Mass,and UV-Vis]. Also molar conductivity, elemental microanalysis, melting point, magnetic sensitiveness, and atomic absorption were applied to portray the synthesized mixed ligand complexes. Some physical properties were listed in Table 1.
3.1. FT-IR Spectra

In the spectra of free [HQ] ligand is found the peak found at 3182 cm$^{-1}$ assigned to the alcohol $\nu$(OH) stretching and this band was absented in the complexes[12]. In the spectrum of ligand [HL] the sharp peak found at 3458 cm$^{-1}$ assigned to the $\nu$(OH) stretching of carboxylic acid while the band was disappeared in the complexes, proton of ligand [HL]on complexation and participation of hydroxyl hydroxylic anionic (COO$^-$) oxygen in concert [13]. In addition the two peaks at found in range (3468-3455)cm$^{-1}$ and (3387-3348)cm$^{-1}$assigned to stretching of usym and asy(NH$_2$), in the ligand and all the mixed complexes [14]. Also the band found at (1664) cm$^{-1}$is assigned to $\nu$(C=O) group of carboxylic acid of the ligand [HL] [15], this band disappeared in all the mixed complexes In the spectra of the mixed complexes, another bands appeared in range(1589-1572) cm$^{-1}$ and (1465-1445) cm$^{-1}$due to $\nu$asym.(COO$^-$) and $\nu$sym.(COO$^-$) vibration, respectively, ($\Delta$u(COO$^-$)=$\nu$asym.(COO$^-$)−$\nu$sym.(COO$^-$)) in range (116-127)cm$^{-1}$, indicated the involvement of deprotonated group of (COOH) in bonding and a coordination of metal ions through oxygen atoms of carboxyl groups[16]. In the ligand [HL] spectrum was exhibited the strong band at 1637cm$^{-1}$due to $\nu$(C=N) stretching vibration, while this bond was shifted towards lower region at (1624-1627) cm$^{-1}$ in the complexes indicating the participating of the azomethine group in the complex formation. Further, the bands observed at (1591) cm$^{-1}$ and (1573) cm$^{-1}$which were due to $\nu$(C=N) azomethine groups in rings for ligand free [HL] and ligand free [HQ], while these bands were shifted in range(1566-1553)cm$^{-1}$ and (1523-1492)cm$^{-1}$due to the reduction of double bond character carbon nitrogen bond of azomethine group and indicates that (C=N) of the ligand co-ordinate to metal through nitrogen and that was further reflected by the appearance of new bands at (586-405)cm$^{-1}$due to $\nu$(M-N)[17]. The mixed ligand complexes formation was further evidence by the appearance of the ligand band in the complexes (455-405)cm$^{-1}$ assigned as $\nu$(M-O) bonds[18]. In the IR spectra of mixed ligand complexes a band was observed around (3356-3197) cm$^{-1}$ to which were assigned to hydrate water molecules [19, 24].
Table 2. The FT-IR spectral data (cm\(^{-1}\)) of all the prepared compounds

| Compound           | \(\nu(\text{OH})\) | \(\nu(\text{H}_2\text{O})\) | \(\nu(\text{N-H})\) | \(\nu(\text{C=N})\) | \(\nu(\text{C=O})\) | \(\nu(\text{COO})\) | \(\Delta \nu\) | \(\nu(\text{M-\text{OH})}\) | \(\nu(\text{M-N})\) | \(\nu(\text{M-O})\) |
|--------------------|----------------|----------------|-----------------|----------------|----------------|----------------|----------|----------------|----------------|----------------|
| [HL]               | 3329           | 3468           | 1591            | 1637           | 1664           | -              | -        | -              | -              | -              |
|                    | 3388           |                |                 |                |                |                |          |                |                |                |
| [HQ]               | 3356           | 3468           | 1553            | 1625           | -              | 1572           | 1445     | 127            | 127            | 513            | 451           |
|                    | 3387           | 1523           |                 |                |                |                |          |                |                |                | 482           |
| [Mn(L)Q(H\(_2\)O)] | -              | 3197           | 1562            | 1624           | -              | 1585           | 1462     | 123            | 123            | 558            | 432           |
|                    | 3369           | 1496           |                 |                |                |                |          |                |                |                | 497           |
| [Co(L)Q(H\(_2\)O)] | -              | 3348           | 1558            | 1627           | -              | 1581           | 1465     | 116            | 116            | 547            | 451           |
|                    | 3352           | 1500           |                 |                |                |                |          |                |                |                | 497           |
| [Ni(L)Q(H\(_2\)O)] | -              | 3321           | 1562            | 1625           | -              | 1589           | 1462     | 128            | 128            | 586            | 443           |
|                    | 3385           | 1500           |                 |                |                |                |          |                |                |                | 559           | 408           |
| [Cu(L)Q(H\(_2\)O)] | -              | 3217           | 1558            | 1627           | -              | 1581           | 1465     | 116            | 116            | 504            | 455           |
|                    | 3348           | 1500           |                 |                |                |                |          |                |                |                | 482           | 405           |
| [Zn(L)Q(H\(_2\)O)] | -              | 3332           | 1566            | 1624           | -              | 1577           | 1458     | 120            | 120            | 493            | 439           |
|                    | 3353           | 1492           |                 |                |                |                |          |                |                |                | 455           | 405           |

3.2. NMR Spectra

\(^1\)H-NMR spectrum of the ligand [HL] was recorded in d6-DMSO. The 1H-NMR spectra of [HL] exhibits signal due to proton of (-COOH) group as singlet at \(\delta 12.17\) ppm[20]. The protons of (NH\(_2\)) group as signal at \(\delta 6.62\) ppm[13]. The proton of (-CH-N) pyrimidine ring as singlet at \(\delta 7.15\) ppm. The aromatic protons as multiple at (7.97-7.27) ppm. The (OCH\(_3\)) protons as signal at \(\delta 3.76\) ppm. The DMSO signal appeared at 2.5 ppm [21], figure (3).\(^{13}\)C NMR spectrum of ligand [HL] displays chemical shifts at (170.2) ppm and (164.1) ppm indicate to (C=N) and C=O groups respectively[22]. At (157.4) ppm and (153.4) ppm were the chemical shifts attributed to C atoms of (C=C) in pyrimidine ring, respectively. Signals attached to aromatic carbon were attached in reign (106.7-138.5) ppm. The two chemical shift at (56.6) ppm and (39.6) ppm were referred to C atoms, respectively. Also the chemical shift at (40.2) ppm is due to DMSO d\(_6\)[23], figure 4.'
3.3. Mass Spectra
Mass Spectrum was completed on the ligand [HL] to determine its molecular weight and fragmentation pattern, Figure 5.
3.4. Electronic spectra, Magnetic moments and conductivity measurements

The electronic spectrum of ligand [HL] exhibit high intense absorption peaks at (279 nm) (35842 cm\(^{-1}\)) and (345 nm) (28985 cm\(^{-1}\)) which due to (\(\pi \rightarrow \pi^*\)) and (n\(\rightarrow \pi^*\)) transitions, respectively [24], the data recorded in Table (3). The electronic spectrum of 8-hydroxy quinolone [HQ], displays high intense absorption bands at (268 nm) (37313 cm\(^{-1}\)) and (305 nm) (3278 6cm\(^{-1}\)) which designated to (\(\pi \rightarrow \pi^*\)) and (n\(\rightarrow \pi^*\)) transition respectively [25], the data recorded in Table (3). Theses transitions were also found in the spectra of the complexes, but they were shifted towards in range (37037-14265) cm\(^{-1}\). The electronic spectrum of [Mn(L)(Q)(H\(_2\)O)] showed two intense peaks in (270nm) (37037 cm\(^{-1}\)) and (374nm) (2(737cm\(^{-1}\)) was assigned to the ligand field respectively. And another intense peak in (392nm) (25510 cm\(^{-1}\)) due charge transfer transition. And the peak at visible region at (755 nm) (13245cm\(^{-1}\)). This peak is assigned to (\(^{4}A_{1g} \rightarrow ^{4}T_{2g}(G))\) (d-d) transitions confirming an octahedral structure around (Mn\(^{2+}\)) ion complex[26]. The electronic spectrum of [Co(L)(Q)(H\(_2\)O)] displayed two intense peaks in (277nm) (36101 cm\(^{-1}\)) and (334 nm) (29940 cm\(^{-1}\)) were assigned to the ligand field. And another intense peak in (401 nm) (24937cm\(^{-1}\)) due charge transfer transition. And the peaks at visible region at (687 nm)(14556cm\(^{-1}\)) and (762nm)(13123cm\(^{-1}\)).These peaks are assigned to (\(^{4}T_{1g} \rightarrow ^{4}A_{1g})\) and (\(^{4}T_{1g} \rightarrow ^{4}T_{2g}(G))\) (d-d) transitions confirming an octahedral structure around (Co\(^{3+}\)) ion complex[27]. The electronic spectrum of [Ni(L)(Q)(H\(_2\)O)] showed two intense peaks in (275nm) (36363cm\(^{-1}\)) and (345nm) (28985cm-1) was assigned to the ligand field respectively. And another intense peak in (408nm) (24509 cm-1) due charge transfer transition. And the peak at visible region at (810 nm)(12345 cm\(^{-1}\)). These peaks are assigned to (\(^{4}A_{1g} \rightarrow ^{4}T_{1g}(d-d))\) transitions confirming an octahedral structure around (Ni\(^{2+}\)) ion complex[28]. The electronic spectrum of [Cu(L)(Q)(H\(_2\)O)] showed two intense peaks in (276nm) (36231 cm\(^{-1}\)) and (337nm) (29673cm\(^{-1}\)) was assigned to the ligand field respectively. And another intense peak in (412nm) (24271cm\(^{-1}\)) due charge transfer transition. And the peaks at visible region at (701 nm)(14265cm\(^{-1}\)) and (859nm)(11641cm\(^{-1}\)).These peaks are assigned to (\(^{4}B_{1g} \rightarrow ^{4}B_{2g})\) and (\(^{4}B_{1g} \rightarrow ^{4}A_{1g}(d-d))\) transitions confirming an octahedral structure around (Cu\(^{2+}\)) ion complex[29]. The electronic spectrum of [Zn(L)(Q)(H\(_2\)O)] showed absorption peaks in (277 nm) (36101cm\(^{-1}\)) and (340 nm) (29411cm\(^{-1}\)) were assigned to the ligand field. And another peak in (419 nm) (23866 cm\(^{-1}\)) due charge transfer transition. The absence of absorption peaks at the visible area suggested n(d-d)electronic transitions happened, this is a very well result for an octahedral structure around (Zn\(^{2+}\)) ion complex[30,31]. The electronic spectrum of [Cd(L)(Q)(H\(_2\)O)] showed absorption peaks in (277 nm) (36101cm\(^{-1}\)) and (342nm) (29239 cm\(^{-1}\)) were assigned to the ligand field. And another intense peak in (412 nm) (24271 cm\(^{-1}\)) due charge transfer transition. The disappeared of peaks at the visible area suggested n(d-d)electronic transition same, this is a good result for an octahedral structure around (Cd\(^{2+}\)) ion complex [30, 31]. Where a rapprochement between the data recorded of all the prepared compounds are given in Table 3. The molar conductivities indicate that all metal complexes are non-electrolytes [32], Table 3. Magnetic moment together with these values suggest octahedral configuration around the Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) metal ions studied, Table 3.
Table 3. UV-Vis spectral and magnetic moments values of the compounds in DMSO

| Compounds | λ nm | ε max (molar \cdot cm^{-1}) | Transitions | μ eff (BM) |
|-----------|------|----------------------------|-------------|-----------|
| [HL]      | 279  | 35842                      | 2111        | n→π*      | -         |
|           | 345  | 28985                      | 2260        | n→n*      | -         |
| [HQ]      | 268  | 37313                      | 861         | n→n*      | -         |
|           | 305  | 32786                      | 1966        | n→n*      | -         |
| [Mn(L)(Q)(H₂O)] | 270 | 37037                      | 1675        | L F       | 5.72      |
|           | 374  | 26737                      | 297         | C T       |           |
|           | 392  | 25510                      | 10          |           |           |
|           | 755  | 13245                      |             |           |           |
| [Co(L)(Q)(H₂O)] | 277 | 36101                      | 2400        | L F       | 4.72      |
|           | 334  | 29940                      | 1508        | C T       |           |
|           | 401  | 24837                      | 6           | L F       |           |
|           | 687  | 14556                      |             |           |           |
|           | 762  | 13123                      |             |           |           |
| [Ni(L)(Q)(H₂O)] | 275 | 36363                      | 2315        | L F       | 3.38      |
|           | 345  | 28985                      | 1339        | C T       |           |
|           | 408  | 24509                      |             | 3 A Ig    |           |
|           | 810  | 12345                      |             |           |           |
| [Cu(L)(Q)(H₂O)] | 276 | 37453                      | 2329        | L F       | 1.81      |
|           | 337  | 29673                      | 995         | C T       |           |
|           | 412  | 24271                      | 5           | 3 B g     |           |
|           | 701  | 14265                      |             |           |           |
|           | 859  | 11641                      |             |           |           |
| [Zn(H₂L)(Q)(H₂O)] | 277 | 36101                      | 2463        | L F       | Dia       |
|           | 340  | 41666                      | 1687        | C T       |           |
|           | 419  | 23866                      |             |           |           |
| [Cd(L)(Q)(H₂O)] | 277 | 36101                      | 2418        | L F       | Dia       |
|           | 342  | 29239                      | 1368        |           |           |
|           | 412  | 24271                      |             |           |           |

Dia = Diamagnetic

3.5. Antibacterial Activity Studies:

Finally, the in vitro antibacterial activities of the ligands and their complexes were tested against (Staphylococcus aureus), (Escherichia coli), (Enterobacter cloacae) and (Bacillus subtilis) using well diffusion method by nutrient agar as medium at (10⁻³) mole/liter concentration was prepared by dissolving the compound in DMSO[33]. The zone of inhibition of bacterial evolution around the disc is offered in Figure 6, Table 4 displays the obstruction capacity versus the bacteria sample of the synthesized compounds under work.

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Table 4 the obstruction capacity versus the bacteria sample of the synthesized compounds under work.
Table 1. List of Compounds and their Antimicrobial Activity

| No. | Compound          | Staphylococcus aureus | Bacillus subtilis | Enterobacter cloacae | Escherichia Coli |
|-----|-------------------|-----------------------|-------------------|----------------------|------------------|
| 1   | [HL]              | 43                    | 34                | -                    | 31               |
| 2   | [HQ]              | 32                    | 28                | 25                   | 26               |
| 3   | [Mn(L)(Q)(H_2O)]  | 26                    | 24                | -                    | 22               |
| 4   | [Co(L)(Q)(H_2O)]  | 26                    | 24                | -                    | 24               |
| 5   | [Ni(L)(Q)(H_2O)]  | 31                    | 28                | 13                   | 26               |
| 6   | [Cu(L)(Q)(H_2O)]  | 34                    | 33                | 16                   | 30               |
| 7   | [Zn(L)(Q)(H_2O)]  | 30                    | 33                | 25                   | 28               |
| 8   | [Cd(L)(Q)(H_2O)]  | 27                    | 30                | 22                   | 27               |
| C   | DMSO              | -                     | -                 | -                    | -                |

Figure 6. Shows the antimicrobial activity of compounds appear the inhibition zones against pathogenic bacteria (Staphylococcus Aureus, Bacillus Subtilis, Enterobacter Cloacae and Escherichia Coli)

4. Conclusion

Mixed ligand complexes can be a synthetic challenge to tune the properties of the metal complexes and have been shown to exhibit a broad range of the possible geometry of synthesized complexes is octahedral and it is six coordinated metal ligand complexes. The ligand Schiff base [HL], ligand [HQ] and their metal complexes showed broad-spectrum antimicrobial activities against all the microbes used.

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