**INTRODUCTION**

The imidazole and thiazole derivatives characterized ligands azo dyes of heterocyclic compounds as highly effective against most of the elements of the periodic table as chemistry complexes. These ligands from a wide field of in practice. It is well known that for this type of compounds of the importance in the biological field due to presence of these compounds on different atoms such as oxygen, nitrogen and sulfur−qualify it for the link with the different elements and that small traces of these elements leads to inhibition of vitality activities. Using azo imidazole and thiazolyl azo in different fields have been used in medicine, science and technology, giving results are of great importance in the life.

A new ligand containing thiazole and imidazole rings as organic heterocyclic azo dye compound 2-[2-(6-nitro benzothiazolyl)azo]imidazole (NBTAI) was prepared by coupling reaction between 6-nitro benzothiazole chloride with imidazole in alkaline alcoholic solution. New six complexes with Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) were prepared and characterized by elemental analysis, molar conductance measurements, magnetic moment measurements at room temperature, infrared and electronic spectra. The analytical data show that the metal to ligand ratio [M:L] in all complexes is (1:2). The spectral and analytical data revealed that this ligand behaves as a bidentate chelating agent and the coordination number of all metal ions are found to be six in coordination through the nitrogen N3 atom of azo methine group of heterocyclic imidazole ring and the other nitrogen atom of azo group which is the farthest of imidazole molecule, from two ligands with two molecules and two chloride ions. The results are explained in the light of these analysis.

Key Words: Azo ligand, Coupling reaction, Metal complexes, Spectroscopic studies.

**EXPERIMENTAL**

All chemical and solvents were purchased from Aldrich, Sigma, Fluka and B.D.H, except of 2-amino-6-nitro benzothiazole was prepared as described in the literature. All chemical have been used without further purification and used as supplied by the companies. IR spectra were recorded as KBr discs using a Shimadzu 8400FTIR spectrophotometer in range (4000-400) cm\(^{-1}\). Electronic spectra of the prepared ligand and its complexes were measured in the region (200-1100) nm by using ethanol as solvent at 25 °C using Shimadzu UV-visible. 1650 pc spectrophotometer, with 1,000 ± 0.001 cm\(^{-1}\) match quartz cell. CHNS elemental analysis was performed on a Euro EA elemental analyzer (Al-Kufa University). The magnetic susceptibilities were measured on a MSB-Auto Magnetic Susceptibility Blance (Al-Nahrain University). Electrical conductivity measurements of the chelate complexes were recorded at room
Preparation of the azo dye ligand (NBTAI): The heterocyclic azo ligand (NBTAI) was prepared by the diazotization coupling reaction by following method proposed by Shibata et al.\textsuperscript{23} with some modifications (Scheme-I). 2-Amino-6-nitro benzothiazole (1.95 g, 0.01 mol) was dissolved in mixture 30 mL distilled water and 5mL of concentrated hydrochloric acid. The filtered solution was diazotized at 0-5 °C with (0.84 g, 0.012 mol) sodium nitrite was dissolved in mixture 30 mL distilled water plus 15 mL of acetic acid-propionic acid mixture (15 mL acetic acid plus 5 mL of propionic acid), was added drop wise to solution of 2-amino-6-nitro benzothiazole and stirred for 25 min at 0-5 °C in ice-bath. The resulting diazonium chloride solution was added dropwise with cooling and stirring continuously at 0-5 °C. The imidazole (0.68 g, 0.01 mol) was dissolved in 150 mL of ethanol and 50 mL of 7 % sodium hydroxide solution and 50 mL of 7 % sodium carbonate solution were added. The mixture was stirred continuously for 3 h at 0-5 °C in ice-bath and allowed to stand overnight and concentrated (rotary evaporator). The precipitate was filtered and washed several times with cold distilled water until a negative chloride reaction with silver nitrate as well as washed with mixture distilled water and ethanol solution to remove the excess of unreacted substances. The product was finally recrystallized from (1 + 1) aqueous ethanolic solution and dried in oven at 60 °C for several hours and stored in a desiccator over anhydrous calcium chloride. The yield was 78 % of orange powered. The purity was confirmed by the elemental analysis and TLC techniques.

Preparation of metal complexes: The metal complexes were prepared by adding 0.629 g, (0.002 mol) of ligand (NBTAI) dissolved in hot ethanol (70 mL) and added dropwise with stirring a stoichiometric amount of 1:2 [M:L] molar ratio (0.001 mol) for Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) chloride salt dissolved in 40 mL hot buffer solution ammonium acetate at optimal pH for each metal ions. The reaction mixture was heated to 50-60 °C for 30-40 min, until solid complexes were precipitated then left over night. The solid product thus formed was filtered off, washed distilled water until the solution become colourless and washed with 10 mL hot ethanol to remove any traces of the unreacted materials and dried in a desiccators over anhydrous CaCl\textsubscript{2}. Table-1 shows the collects the physical properties and analytical data for the prepared ligand and their complexes.

RESULTS AND DISCUSSION

Characterization of azo dye ligand and its metal complexes: The azo dye ligand (NBTAI) was orange powder but the obtained complexes were found to be different colour crystals depending on the nature metal ion and stable in air at room temperature. The ligand and its chelate complexes were insoluble in water but soluble in most organic solvents such as ethanol, methanol, acetone, DMF and DMSO. The purity of azo dye complexes were tested by TLC technique and C.H.N.S elemental analysis.

Effect of pH: The effect of pH values for metal complex solutions was adjusted using dilute solutions (0.1 M) of ammonium acetate, acetic acid and ammonia solution and the effect on absorbance was studied to determine the optimum pH in each case. The absorption of all chelate complexes solutions did not change over the whole range and very stable at pH = 6-8. The result are shown in Figs. 1 and 2 and maximum absorbance was obtained in pH range given in Table-1.

Effect of reaction time: The effect of time to get the complex is an important factor to obtain knowledge of the time period maintains the complex on stability especially employed in the field of analytical chemistry. The stabilizing complexes

\begin{equation}
\text{O}_2\text{N} \begin{array}{c} \text{S} \\
\text{NH}_2 \end{array} \text{N} \quad \begin{array}{c} \text{NaNO}_2 \\
\text{Distilled water + propionic acid + acetic acid} \end{array} \text{O}_2\text{N} \begin{array}{c} \text{S} \\
\text{N} \end{array} \text{NCl} \\
\text{Diazonium salt} \end{equation}

2-Amino-6-nitro benzothiazole distilled water + conc. HCl

\begin{equation}
\text{O}_2\text{N} \begin{array}{c} \text{S} \\
\text{N} \end{array} \text{N} \quad \begin{array}{c} \text{H} \\
\text{In ethanol, pH = 6.0} \end{array} \\
\text{2-[2-(6Nitro benzothiazolyl)azo]imidazole} \quad \begin{array}{c} \text{N} \\
\text{NBTAI} \end{array} \end{equation}

\begin{equation}
\text{O}_2\text{N} \begin{array}{c} \text{S} \\
\text{N} \end{array} \text{N} \quad \begin{array}{c} \text{H} \\
\text{In ethanol, pH = 6.0} \end{array} \\
\text{7 % NaOH + 7 % Na}_2\text{CO}_3 \end{equation}

Scheme-I: Preparation of azo dye ligand2-[2-(6-nitro benzothiazolyl)azo]imidazole (NBTAI)
preparation of complex solution access to 180 min. The results are described in the Figs. 3 and 4 indicating for the moment of the preparation of complex solution access absorption for solutions complexes for a period of time ranged absorption (λ_max).

The solutions of prepared complexes have been studied at the maximum wavelength absorption (λ_max) per complex by continue in the change at absorption for solutions complexes for a period of time ranged for the moment of the preparation of complex solution access to 180 min. The results are described in the Figs. 3 and 4 indicate that the stability of solution of these complexes over time 10-15 min from the moment you start mixing which useful researcher in the field of spectral determination of metal ions. The reaction complete in 5 min at room temperature and remains stable for at least 24 h. This show the azo dye ligand (NBTAI) demonstrates strong coordination ligand with all metal ions.

\[ \text{LH} = (\text{NBTAI}) \]

### TABLE-1

| Compound       | m.p. (°C) | pH | Colour         | Yield (%) | m.f. (m.w.) | C       | H       | N       | S       | M      | Found Calc. (%) |
|----------------|-----------|----|----------------|-----------|-------------|---------|---------|---------|---------|-------|-----------------|
| LH = NBTAI     | 168       | 6.0| Orange         | 78        | C_1H_3N_3S_2O_2 | 43.61   | 2.15    | 30.72   | 11.52   | –     | –               |
| [Co(L)_2Cl_3]·H_2O | 201      | 7.5| Green          | 71        | C_2H_8N_3S_2O_2ClCo | 34.61   | 1.96    | 24.02   | 9.05    | 8.29  | –               |
| [Ni(L)_2Cl_3]  | 187       | 7.0| Reenish blue   | 64        | C_2H_8N_3S_2O_2ClNi | 35.24   | 1.72    | 24.61   | 9.26    | 8.47  | –               |
| [Cu(L)_2Cl_3]·H_2O | 213     | 6.5| Green          | 76        | C_2H_8N_3S_2O_2ClCu | 34.15   | 1.92    | 23.89   | 9.39    | 8.86  | –               |
| [Zn(L)_2Cl_3]  | 192       | 7.0| Purple         | 57        | C_2H_8N_3S_2O_2ClZn | 35.19   | 1.69    | 24.41   | 9.53    | 9.37  | –               |
| [Cd(L)_2Cl_3]·H_2O | 203     | 6.0| Reddish purple| 53        | C_2H_8N_3S_2O_2ClCd | 32.13   | 1.83    | 22.56   | 8.76    | –     | –               |
| [Hg(L)_2Cl_3]·H_2O | 208     | 8.0| Dark purple    | 68        | C_2H_8N_3S_2O_2ClHg | 28.48   | 1.61    | 20.19   | 7.88    | –     | –               |

Fig. 1. Effect of pH on absorbance values of chelate complexes

Fig. 2. Effect of pH on absorbance values of chelate complexes

Fig. 3. Effect time on stability Ni(II), Co(II) and Cu(II)-complexes

Fig. 4. Effect time on stability Zn(II), Cd(II) and Hg(II) -complexes

Metal:ligand ratio: For the purpose of finding possible structural formula of prepared complexes was studied by molar ratio method at fixed optimum pH and concentration at wavelength maximum absorption (λ_max). The solutions of prepared...
complexes increase the intensity of the colours as approach point of intersection ratio (M:L) and color continuous constant at passing this point which indicates that the complex formed in constant of solution. A 1:2 [M:L] mole ratio suggested the formation of [M (NBTAI)] where M = Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II).

**Calculation of the metal complexes stability constant:**
The calculation of stability constants of complexes in their solutions depending on spectroscopic methods, particularly if colored complexes have taking advantage of the absorbance values obtained from calculations molar ratio of chelate complexes. The stability constants are obtained by measuring the absorbance of solution mixture of ligand and metal ion at fixed wavelength ($\lambda_{\text{max}}$) and optimum pH values. The degree of formation of the complexes is calculated according to the relation $\beta = (1-\alpha/4\alpha^2)$, where $\alpha = (Am-As/Am)$, which means it does not coordination the metal ion with nitrogen of thiazole ring. One absorption band is observed at 1459 cm$^{-1}$ in free ligand spectrum is assigned to the existence of N(N=N) aza group while in chelate complexes spectra these bands are shifted to lower frequencies at 1435-1415 cm$^{-1}$ with decreased or increased intensity.

The changing of position and intensity of this absorption band (aza group) indicates the effect of this group and by coordination. Another bands appeared at 1335 and 840 cm$^{-1}$ in the free ligand spectrum. These bands are due to v(C-S) of the thiazole ring. The existence of these bands in all metal complexes means that the sulphur atom of the thiazole ring does not participate in coordination. New absorption in the range of 520-447 cm$^{-1}$ which are not present in the spectrum of free ligand are due to $v$(M-N) band vibration. IR spectra data lead to suggest that the ligand behaves as a bidentate chelating agent coordinating through the nitrogen atom of aza group nearest to thiazole ring and N$_1$ atom of imidazole ring to give five membered chelate ring. Some representative spectra are given in Fig. 5-7.

**Molar conductivity measurements:** The studied of molar conductivity of preapred chelates solid solutions for ligand (NBTAI) were carried out in ethanol and DMF solvents (10$^{-2}$ M at room temperature. The results proved in the Table-2 this study has shown the values of molar conductivity of all metal complexes that non-electrolyte and no conductive species exist.

**Infrared spectra:** The infrared absorption of the ligand its complexes (KBr disc) have been studied and given in Table-3. The comparison between spectra of ligand with the coordination complexes have revealed certain characteristic differences. The IR spectrum of free ligand (NBTAI) shows a weak and broad band around 3437 cm$^{-1}$ due to $v$(N-H) of imidazole ring. This band remains in the same region in ligand and in solid chelate complexes spectra. The remaining of the hydrogen of (N-H) group indicates non involvement in coordination of the ligand to the metal ions. IR spectra of Co(II), Cu(II), Cd(II) and Hg(II) metal complexes exhibit a broad in the range of 3332-3280 cm$^{-1}$ due to presence of water molecules. Two weak absorption bands had been observed at 3085 cm$^{-1}$ and 2970 cm$^{-1}$ in ligand spectrum which are due to $v$(C-H) aromatic and aliphatic respectively. These bands are stable in position and intensity for both free ligand and metal complexes. The ligand shows two absorption bands at 1615 cm$^{-1}$ and 1518 cm$^{-1}$ due to $v$(C=N) of thiazole and imidazole rings. In the spectra of metal complexes it is observed with allitie change in shape and shifted to lower frequencies at 1517-1490 cm$^{-1}$ attributable $v$(C=N) of imidazole ring, while the remaining absorption band $v$(C=N) attributable to thiazole ring is stable in position and unchanged in all metal complexes which means it does not coordination the metal ion with nitrogen of thiazole ring. One absorption band is observed at 1459 cm$^{-1}$ in free ligand spectrum is assigned to the existence of N(N=N) aza group while in chelate complexes spectra these bands are shifted to lower frequencies at 1435-1415 cm$^{-1}$ with decreased or increased intensity.

**Electronic spectra and magnetic moments:** The electronic spectra of ligand and their metal complexes were studied in absolute ethanal as a solvent. The spectral data and magnetic moments are listed in Table-4 and Fig. 8-11. The free ligand (NBTAI) spectrum gives three absorption bands were detected.
first band located at 387 nm (25840 cm\(^{-1}\)) for \(n\rightarrow\pi^*\) transition of the azo group (N=N), this band showed a red shift on coordination with a metal ion. The second band observed at 291 nm (34364 cm\(^{-1}\)) due to \(\pi\rightarrow\pi^*\) transition to the \(C=\) group in heterocyclic imidazole and thiazole rings. While the third band at 263 nm (38023 cm\(^{-1}\)) presents a blue shift for \((n\rightarrow\sigma)\) transition with these rings.

**Cobalt(II)-complex:** The electronic spectrum of Co(II)-complex was studied in \(10^{-3}\) M ethanolic solution shows three absorption bands at 974 nm (10235 cm\(^{-1}\)), 629 nm (15898 cm\(^{-1}\)) and 350 nm (26571 cm\(^{-1}\)). These are assigned to \(^4T_{1g(F)}\rightarrow^4T_{2g(F)}\) \((\nu_1)\), \(^4T_{1g(F)}\rightarrow^4A_{2g(F)}\) \((\nu_2)\) and \(^4T_{1g(F)}\rightarrow^4T_{2g(F)}\) \((\nu_3)\) transition, respectively. The magnetic moment of this complex has been found to be 5.07 B.M which correspond to three unpaired electrons which may suggest an distorted octahedral structure (Z-out) and hybridization \(sp^d^2\) \((^3\Sigma_g\ ^2\Sigma_g^+\ ^2\Pi_g)\).

**Nickel(II)-complex:** The electronic spectrum of this complex show three absorption bands in \(10^{-3}\) M ethanolic solution at 977 nm (10235 cm\(^{-1}\)), 636 nm (15723 cm\(^{-1}\)), which suggesting the existence of \(^3A_{2g}\rightarrow^3T_{2g(F)}\) \((\nu_1)\), \(^3A_{2g}\rightarrow^3T_{1g(F)}\) \((\nu_2)\) and \(^3A_{2g}\rightarrow^3T_{1g(F)}\) \((\nu_3)\) transition respectively. For the Ni(II)-complex the value of magnetic moment was found to be 3.18 B.M. because of presence of two unpaired electrons which may suggest a high spin a regular octahedral geometry \(^{6}\) \((^2\Sigma_g\ ^2\Pi_g)\) and hybridization \(sp^d^2\).

**Copper(II)-complex:** The electronic absorption spectrum of Cu(II)-complex shows a broad band around at 623 nm (16051 cm\(^{-1}\)) in \(10^{-3}\) M ethanolic solution due to \(^2E_g\rightarrow^2T_{2g}\)

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**TABLE-3**

| Compound                  | \(v(N-H)\) | \(v(O-H)\) | \(v(C-N)\) imidazoles | \(v(C-N)\) thiazole | \(v(N=N)\) | \(v(C=C)\) | \(v(C-N=N-C)\) | \(v(C=S)\) thiazole | \(v(C-N)\) thia. | \(v(M-N)\) |
|---------------------------|-------------|-------------|------------------------|---------------------|-------------|-------------|-------------------|---------------------|------------------|-------------|
| LH=NBTAI                  | 3437w, br.  | –           | 1615m.                 | 1518s.              | 1459m.      | 1335s.      | 1245w.           | 1132w.              | 867m.            | 1107m.      |
| [Co(L)\(_2\)Cl\(_2\)]H\(_2\)O | 3427w, br.  | 3420w, br. | 1590m.                 | 1515m.              | 1440m.      | 1400 m, h. | 1255w.           | 1185s.              | 860m.            | 1120s.      |
| [Ni(L)\(_2\)Cl\(_2\)]     | 3418w, br.  | –           | 1589m.                 | 1519vs.             | 1448w.      | 1342vs.     | 1240w, sh.       | 1140w, br.          | 856m.            | 1103m, sh. |
| [Cu(L)\(_2\)Cl\(_2\)]H\(_2\)O| 3394m.      | 3332w, br. | 1658m.                 | 1512m.              | 1434m.      | 1342m.      | 1265w.           | 841m.               | 1242m.          | 987m.       |
| [Zn(L)\(_2\)Cl\(_2\)]     | 3440 –      | 1595m.      | 1520s.                 | 1432m.              | 1378s.      | 1245m, sh.  | 1128m.           | 855m.               | 1109m.          | 985m, sh.  |
| [Cd(L)\(_2\)Cl\(_2\)]H\(_2\)O| 3432 –      | 3417w, br. | 1593m.                 | 1523s.              | 1421m.      | 1337m.      | 1240m, sh.       | 1130m.              | 847m.            | 1115m, sh. |
| [Hg(L)\(_2\)Cl\(_2\)]H\(_2\)O| 3448 –      | 3360w, br. | 1610vs. s.             | 1520vs.             | 1445vs.     | 1348m.      | 1260v.s.         | 1238m.              | 8845s.          | 985m, sh.  |

Fig. 6. IR spectrum of Co(III)-complex

Fig. 7. IR spectrum of Hg(II)-Complex

Fig. 8. UV-visible spectrum of ligand(NBTAI)

Fig. 9. UV-visible spectrum of Ni(II)-Complex
Zinc(II), cadmium(II) and mercury(II) complexes:
The electronic spectra of Zn(II), Cd(II) and Hg(II) complexes were recorded in 10⁻³ M ethanolic solution, do not show any d-d transitions because they are saturated with electrons (nd¹⁰). The absorption bands at 521 nm (15194 cm⁻¹), 518 nm (15305 cm⁻¹) and 537 nm (16622 cm⁻¹) these are assigned to a charge transfer (M → L, CT) transition to Zn(II), Cd(II) and Hg(II) complexes respectively. The magnetic susceptibility show that these metal complexes have diamagnetic moment (µₑₑ = 0.00) BM and a octahedr al geometry and hybridization sp³d⁵ (³t₂⁺, ⁴e_g⁰)²¹,²².

According to these results and discussed through different techniques suggest below the proposed the structural formula of chelate complexes prepared and shown in Fig. 12.
Conclusion

In this paper, we reported the preparation, spectral identification, analytical data and coordination chemistry of azo dye ligand derived from thiazole and imidazole rings (NBTAI) obtained from the coupling reaction of 6-nitro benzothiazole diazonium chloride salt with imidazole and its metal complexes with Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) metal ions. The azo ligand (NBTAI) behaves as a bidentate chelating agent coordination through the N atom of the imidazole ring and another nitrogen atom of azo group to form five member metal-ring. The geometry is proposed for all complexes octahedral sterochemistry. All complex characterized solid metal complexes not effect by air, light and a moisture, suggesting high stability plus high degrees of melting points that gives another evidence of the stability of prepared metal complexes.

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