

# Synthesis, Characterization and Photophysical Properties of Cu (II), Ni(II), Co(II), and Zn(II) Complexes of Isatin Derivative

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**ABSTRACT:** This research focuses on the preparation, characterization, and the study of photoluminescence properties of new Co(II), Ni(II), Cu(II), and Zn(II) complexes of Isatin Derivatives. The ligand and metal complexes were characterized by spectroscopic studies such as IR, UV/Vis, NMR, photoluminescence, Molecular docking studies. The ligand and the resulting metal complexes gave intense emissions ( $\lambda_{\max} = 267-297$  nm) upon irradiation by ultraviolet light. The photoluminescence intensities and quantum yields of the metal complexes changed upon complexation with various metals ( $\lambda_{\max} = \text{Ni}=361, \text{Zn}=363, \text{Cu}=375, \text{Co}=370$ ) and ( $\lambda_{\max} = \text{Ni}=361, \text{Zn}=351, \text{Cu}=354, \text{Co}=352$ ). Drug designing against breast cancer as they are exhibiting excellent binding property with the BCAR1 protein. These novel complexes may be of interest as organic emitting material for electroluminescent devices.

**KEYWORDS:** Isatin, 2-aminopyridine, physiochemical, photoluminescent

## I.INTRODUCTION

Chemistry of transition metal ions and isatin, 2-aminopyridine derivatives may be an interesting subject for further studies. Schiff bases derived from isatin showed many pharmacological properties and some are most widely used organic compounds. Heterocyclic compounds are of interest in preparative organic synthesis, due to their rich chemistry and biological activities like anticancer[1] antimicrobial[2], anti-malarial[3], and anti-angiogenic[4]and, Isatin also gamely crosses the blood-brain barrier, signifying its possible action on the central nervous system (CNS). Therefore, the study of metal complexes of Schiff-base ligands derived from isatin has received much attention during the last years [5], particularly in the treatment of therapeutic agents, antihypertensive. And also, Pyridine and aminopyridines are well known class of compounds for its potassium channel blocking action.[6]These Schiff bases form an imperative class of the furthestmost widely used organic compounds and has a wide variety of applications in many fields, e.g., biological, inorganic and analytical chemistry[7]. Isatin derivatives has attracted the attention of many experimental and theoretical researches due to their particular photo- luminescence in the visible range and at room temperature andso applied in a large number of realms such as microelectronics, optoelectronics and biological sensors[8-10], photoluminescent heavy metal complexes as emitters in organic light-emitting diodes (OLEDs) have attracted great attention because they can wholly exploit both singlet and triplet excitons through the strong spin-orbital coupling. Transition metals are a growing area of interest due to their important role in biological processes, electrochemistry, and pharmaceutical synthesis[11-14 ]. The goal of the present slog was to synthesize and characterize Co(II), Ni(II), Cu(II), and Zn(II) complexes with Isatin Derivatives. An investigation of the optical properties of the ligand and its various complexes in solution was also carried out. The ligand and its various complexes were found that exhibit intense photoluminescence upon UV irradiation[15-19]. Using gemdock , Drug designing against breast cancer as they are signifying outstanding binding property with the BCAR1 protein[20-22]

# International Journal of Innovative Research in Science, Engineering and Technology

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Vol. 5, Issue 8, August 2016

## II. MATERIALS AND METHODS

All chemicals and solvents used were of LR grade. The proton magnetic spectra were recorded on a Bruker AMX-5000 Spectrometer. IR spectra were recorded on a Jasco FTIR Spectrophotometer. UV-Visible spectra were obtained in DMSO on a Shimadzu UV-Visible Spectrophotometer using Photoluminescence (PL) spectrum of metal complexes under the excitation of light at 220 nm.

### Preparation of Ligand(LH)

Equimolar concentration of Isatin (or) 4-chlorobenzyl isatin (0.01) and 2-Aminopyridine(0.01mol) dissolved in minimum volume of ethanol, and then 2-3 drops of concentrated sulfuric acid are added to the reaction mixture. The solution was stirred for 6hr at 50<sup>0</sup>-60<sup>0</sup>c and the progress of the reaction was monitored by TLC until the reaction was complete. It was cooled to room temperature, the precipitate was filtered washed with ethanol; mp256°C;IR(KBr):( **L1**)  $\nu$  = 3447 (-NH<sub>2</sub>), 3189 (aromatic C-H), 2888 (aliphatic C-H), 1731 s (C=O), 1613-1326 m (C=C and C=N), <sup>1</sup> H-NMR(DMSO):  $\delta$  = 7.00-8.45 (ArH), 7.89 (s, 2H, N-NH<sub>2</sub>), <sup>13</sup>C-NMR(DMSO): (C=O,150),(C=N ,163) (C=C,141) ,Anal. Calcd. for C<sub>13</sub>H<sub>9</sub>N<sub>3</sub>O: C, 69.95; H, 4.06; N, 18.82; O, 7.17. Found: C, 68.93; H, 4.10; N,18.74; O, 7.00%. ) UV/Vis [ $\lambda$  (nm)]: 262, 297. ( **L2**)  $\nu$  = 3434 (-NH<sub>2</sub>), 2954 (aromatic C-H), 2888 ,1608-1344 m (C=C and C=N), <sup>1</sup> H-NMR(DMSO):  $\delta$  = 6.70-7.78 (ArH), 7.88 (s, 2H, N-NH<sub>2</sub>), <sup>13</sup>C-NMR(DMSO): (C=O,160),(C=N ,160) (C=C, 138) Anal. Calcd. for C<sub>20</sub>H<sub>13</sub>ClN<sub>2</sub>O: C, 72.18; H, 3.94; Cl, 10.65 N, 8.42; O, 4.81. Found: C, 72.09; H, 3.88;Cl, 10.61 N,8.34; O, 4.76%. UV/Vis [ $\lambda$  (nm)]: 260, 299.

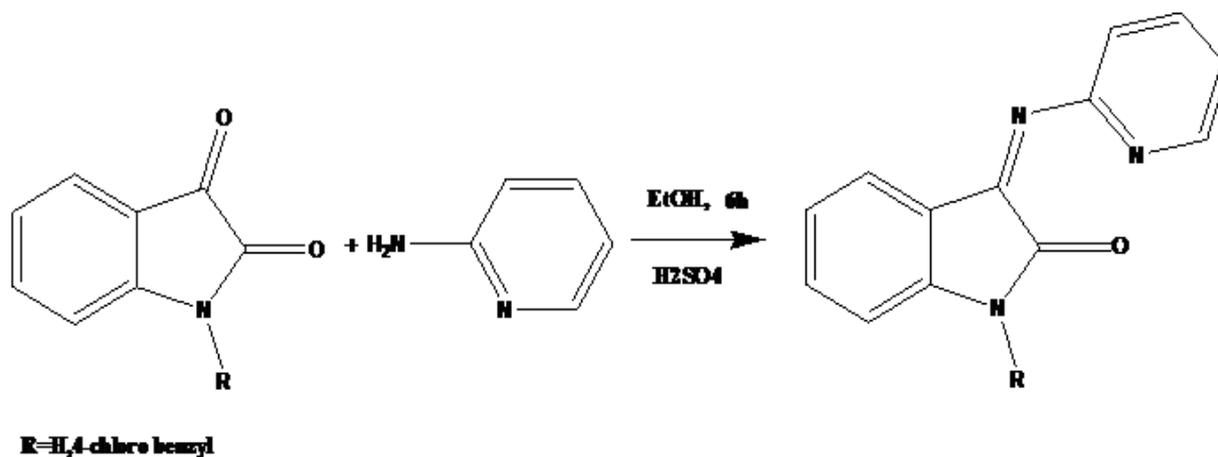


Figure 1. Proposed chemical structure of the ligand

### 3.3 Preparation of Metal complexes

Metal salt (CoCl<sub>2</sub>.2H<sub>2</sub>O, NiCl<sub>2</sub>.6H<sub>2</sub>O, CuCl<sub>2</sub>.2H<sub>2</sub>O and ZnCl<sub>2</sub>.H<sub>2</sub>O) dissolved in hot ethanol (0.001mol) was mixed with hot ethanol solution of the ligand (0.002mol) and refluxed for 1 hr. The complexes separated out in each case. The product was filtered, washed with ethanol and dried under vacuum. The complexes were purified using DMSO- ethanol solvent mixture by recrystallization method.

## International Journal of Innovative Research in Science, Engineering and Technology

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Vol. 5, Issue 8, August 2016

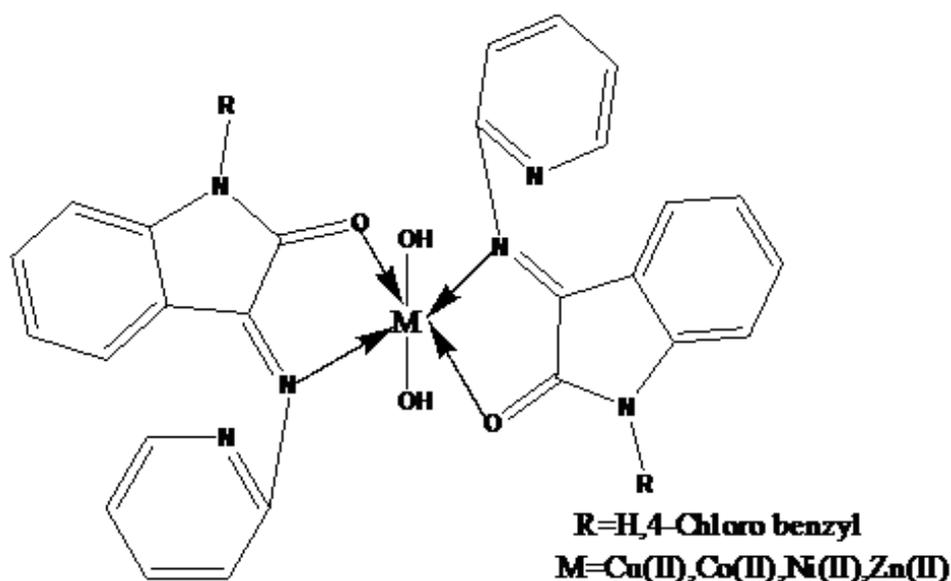


Figure 2. Proposed chemical structure of the Complex

**[CoL1]<sub>2</sub>.H<sub>2</sub>O (1):** Yield was 506 mg (82%), mp 265 °C. Anal. Calcd. for [Co(C<sub>13</sub>H<sub>9</sub>N<sub>3</sub>O)<sub>2</sub>(OH)<sub>2</sub>]·C, 62.58; H, 4.13; Co, 10.97; N, 10.42; O, 11.91. Found: C, 62.50; H, 4.04; Co, 10.93; N, 10.40; Selected IR data (KBr, ν cm<sup>-1</sup>): 3445 ν (OH/H<sub>2</sub>O), 3193 ν (NH), 3 (C=O), 1730 ν (C=O), 1372 (C-N), 1173, (M-O, 550), 452 (M-N). UV/Vis [λ (nm)]: 268

**[NiL1]<sub>2</sub>.H<sub>2</sub>O (2):** Yield was 298 mg (88%), mp 262 °C. Anal. Calcd. for [Ni(C<sub>13</sub>H<sub>9</sub>N<sub>3</sub>O)<sub>2</sub>(OH)<sub>2</sub>]·C, 62.60; H, 4.13; N, 10.43; Ni, 10.93; O, 11.91. Found: C, 62.70; H, 4.03; N, 10.85; O, 11.95%. Selected IR data, ν (cm<sup>-1</sup>): 3401 ν (OH/H<sub>2</sub>O), 3325 ν (NH), 1729 ν (C=O), 1327 (C-N), (M-O, 588), 455 (M-N). <sup>1</sup>H-NMR (DMSO, ppm): δ = 2.0 (m, 1H, O-H) 7.04-7.6 (m, Ar-H), 7.62 (m, Ar-H) 7.5-7.59 (brs, C=C-H), 1.11-3.7 (m, N-H) <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>, ppm), δ 184.36 (OC-Ar), 150.66 (C=N), 140.52 (C=C), 122.74 (C-Ar) 124.64 (C-N)-UV/Vis [λ (nm)]: 267,

**[CuL1]<sub>2</sub>.H<sub>2</sub>O (3):** Yield was 368 mg (76%), mp 234 °C. Anal. Calcd. for [Cu(C<sub>13</sub>H<sub>9</sub>N<sub>3</sub>O)<sub>2</sub>(OH)<sub>2</sub>]·C, 62.04; H, 4.09; Cu, 11.72; N, 10.34; O, 11.81. Found: C, 61.95; H, 4.00; Cu, 11.62; N, 10.28; O, 11.72%. Selected IR data, ν (cm<sup>-1</sup>): 3337 ν (OH/H<sub>2</sub>O), (NH), 3444, 1731 ν (C=O), 1603 (C-N) 1278 (M-O, 590) 453 (M-N). UV/Vis [λ (nm)]: 280.

**[ZnL1]<sub>2</sub>.H<sub>2</sub>O (4):** Yield was 230 mg (68%), mp 251 °C. Anal. Calcd. For [Zn(C<sub>13</sub>H<sub>9</sub>N<sub>3</sub>O)<sub>2</sub>(OH)<sub>2</sub>]·C, 61.83; H, 4.08; N, 10.30; O, 11.77; Zn, 12.02. Found: C, 61.76; H, 4.00; N, 10.28; O, 11.64; Zn, 12.00. Selected IR data, ν (cm<sup>-1</sup>): 3416 ν (OH/H<sub>2</sub>O), 3197 (NH), 1732 ν (C=O), 1327 (C-N), 510 (M-O) ν, 452 (M-N). <sup>1</sup>H-NMR (DMSO, ppm): δ = 2.5 (O-H) 7.6 (Ar-H), 3.2 (NH) 7.5 (C-C-H) <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>, ppm), δ 184.40 (OC-Ar), 150.61 (C=N), 138.34 (C=C), 124 (CN)-UV/Vis [λ (nm)]: 267,

**[CoL2]<sub>2</sub>.H<sub>2</sub>O (5):** Yield was 610 mg (72%), mp 285 °C. Anal. Calcd. For [Co(C<sub>19</sub>H<sub>12</sub>N<sub>3</sub>ClO)<sub>2</sub>(OH)<sub>2</sub>]·C, 69.67; H, 4.38; Co, 8.55; N, 8.12; O, 9.28. Found: C, 69.62; H, 4.34; N, 8.50; O, 9.22. Selected IR data (KBr, ν cm<sup>-1</sup>): 3431 ν (OH/H<sub>2</sub>O), 1735 ν (C=O), 1344 (C-N), 755 (C-Cl) (M-O, 556), 414 (M-N). UV/Vis [λ (nm)]: 297

**[NiL2]<sub>2</sub>.H<sub>2</sub>O (6):** Yield was 580 mg (88%), mp 262 °C. Anal. Calcd. for [Ni(C<sub>19</sub>H<sub>12</sub>N<sub>3</sub>ClO)<sub>2</sub>(OH)<sub>2</sub>]·C, 69.69; H, 4.39; N, 8.13; Ni, 8.13; O, 9.38. Found: C, 69.57; H, 4.43; N, 8.11; Ni, 8.00; O, 9.35%. Selected IR data, ν (cm<sup>-1</sup>): 3437 ν (OH/H<sub>2</sub>O), 1734 ν (C=O), 1343, 755 (C-Cl), (M-O, 542), 416 (M-N). <sup>1</sup>H-NMR (DMSO, ppm): δ = 2.5 (m, 1H, O-H) 7.09-

# International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 8, August 2016

7.6 (m,Ar-H),7.62(m,Ar-H) 7.8(C=C-H).  $^{13}\text{C-NMR}$  (DMSO- $d_6$ , ppm),  $\delta$  182.95(OC-Ar), 150.10(C=N),140.60,(C=C),129.33(C-Ar)124.49(C-N-),39.83(C-Cl)UV/Vis [ $\lambda$ (nm)]: 294,

[CuL<sub>2</sub>]<sub>2</sub> H<sub>2</sub>O (7): Yield was 567 mg (76%), mp 258°C. Anal. Calcd. for [Cu(C<sub>19</sub>H<sub>12</sub>N<sub>3</sub>ClO)<sub>2</sub>(OH)<sub>2</sub>] C, 68.76; H, 4.29; Cu,9.33,N, 8.22; O, 9.39. Found: C, 68.64; H, 4.37; Cu,9.30,N, 8.20; O, 9.33. Selected IR data, $\nu$  (cm<sup>-1</sup>): 3439  $\nu$  (OH/H<sub>2</sub>O), (NH), 1735  $\nu$  (C=O), 1343 (C-N),754 (C-Cl,) 542 (M-O) 414 (M-N). UV/Vis [ $\lambda$  (nm)]: 298.

[ZnL<sub>2</sub>]<sub>2</sub> H<sub>2</sub>O (8): Yield was 598 mg (78%), mp 260 °C. Anal. Calcd. for [Zn(C<sub>19</sub>H<sub>12</sub>N<sub>3</sub>ClO)<sub>2</sub>(OH)<sub>2</sub>] C<sub>38</sub>H<sub>32</sub>N<sub>6</sub>O<sub>3</sub> S<sub>2</sub> Zn (732.00 gmol<sup>-1</sup>): C, 69.02; H, 4.34; N, 8.05; O, 9.19 Zn,9.39. Found: C, 68.00; H, 4.40; N, 8.00; O, 9.00. Selected IR data, $\nu$  (cm<sup>-1</sup>): 3424  $\nu$  (OH/H<sub>2</sub>O), 1735  $\nu$  (C=O), 1334(C-N),756(C-Cl),540(M-O) (415 )M-N).  $^1\text{H-NMR}$  (DMSO, ppm):  $\delta$  = 2.5 (O-H)7.0-7.4 (Ar-H),7.58 (C-C-H),  $^{13}\text{C-NMR}$  (DMSO  $d_6$ ppm),  $\delta$ 184.40 (OCAr), 149.99(C=N), 137.83(C=C), 124.39(C-N-), 41-42(C-Cl) UV/Vis [ $\lambda$ (nm)]: 298,

## III.RESULTS AND DISCUSSION

### Synthesis

Isatin Derivatives (LH, **1**) was prepared according to the literature method by a 2-step synthesis process<sup>20</sup>. The ligand and its complexes were very stable at room temperature in the solid state. The ligand is soluble in common organic solvents, but its metal complexes are generally only soluble in DMF and DMSO.

### Structural Study

#### Physical properties

The Schiff base was freshly prepared by refluxing isatin and 2-aminopyridine (1:1) molar proportion in ethanol in the presence of traces of concentrated sulfuric acid. This Schiff base was then used for the complexation with Cu(II), Co(II), Ni(II), and Zn(II) ions. All of the synthesized metal complexes were air and moisture stable. These were prepared by the stoichiometric reaction of the corresponding metal salts (as chlorides) and the Schiff base in molar ratios M:L of 1:2. The complexes are intensely colored, which decomposes above 2500° C. They are insoluble in common organic solvents such as chloroform or acetone, slightly soluble in ethanol, methanol.

#### Infrared spectra(IR)

Infra-red spectra act as a tool to determine mode of coordination of the ligand to the metal ion. The spectra of the ligand L-1 & L-2 and metal complexes were presented in data. The interpretation of IR spectra of Isatin complexes of transition metal ions helped to confirm the formation of complexes. The IR spectrums of metal complexes were compared with the ligand. The consolidated group frequencies of the chemical bonds of the complexes are represented in which clearly depicts the mode of coordination and complex formation. The stretching vibration modes of free compound Isatin for  $\gamma_{\text{N-H}}$  and  $\gamma_{\text{C=O}}$  bands are shifted from the frequency values 3194 cm<sup>-1</sup> and 1728 cm<sup>-1</sup> to 3439 cm<sup>-1</sup> and 1735 cm<sup>-1</sup> in the newly synthesized ligands. A broad band at 3443 cm<sup>-1</sup> and 3439 cm<sup>-1</sup> is observed due to N-H stretching and it is accompanied by many sub bands appeared in the region 1400-1092 cm<sup>-1</sup> and are associated with N-H in-plane bending. The addition of the organic moiety 4-chlorobenzyl to the Isatin is confirmed shown in the IR spectra of the ligand L-2 the frequency at 756 cm<sup>-1</sup> could be accounted for C-Cl band.

#### UV spectra data

The electronic spectra of the ligands and the complexes were recorded in DMSO as solvent in the UV-Visible region. The electronic transition data was given in the data shows the electronic spectra of the ligands and shows strong absorption bands at 262 nm and 297 nm. These bands may be due to  $\pi \pi^*$ , n  $\pi^*$  transition group of N=C-C=O group. The n  $\pi^*$  transition is due to the presence of lone pair of electrons e<sup>-</sup> on Oxygen atom. Similarly, the ligand B shows strong absorption band at 260 nm, 299 nm due to  $\pi \pi^*$ , n  $\pi^*$  transition group of N=C-C=O group.

## International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 8, August 2016

### <sup>1</sup>H-NMR, <sup>13</sup>C-NMR spectra

The <sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra of the complexes have been recorded for metal complexes. The <sup>1</sup>H NMR spectrum of the complexes shows the signals for the aromatic proton in the range at  $\delta$ -7.0-7.6. In addition, the peak for free O-H group bonding with metal ion is clearly shown at  $\delta$ -2-2.5 for all the complexes. Likewise, it is interesting to observe that the peak for N-H in the range at 11 ppm only for complexes. Substitution of 4-chlorobenzyl without moiety to N-H. Whereas the peak at 11 ppm is absent for the complexes made up of the ligand-2 type. The <sup>13</sup>C NMR spectra of the complexes have been recorded and shown in figure for the metal complexes of Isatin compounds. It is observed that the peak at 184 corresponds to carbonyl carbon shows the signals C=O in the regions of  $\delta$ -182-184, C=N in the regions of

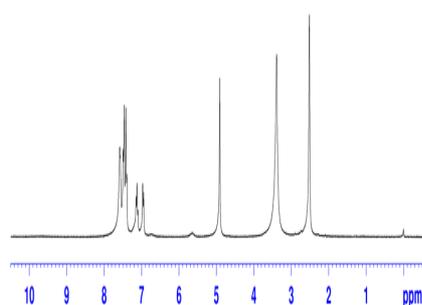


Figure 3. <sup>1</sup>H-NMR Spectrum of Zn(II) complex

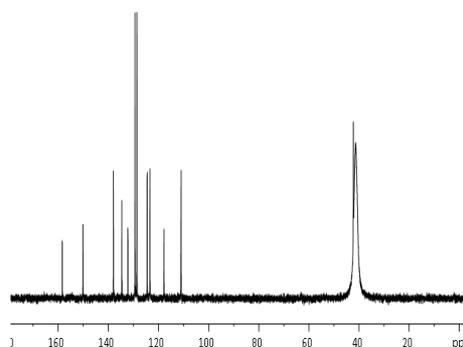
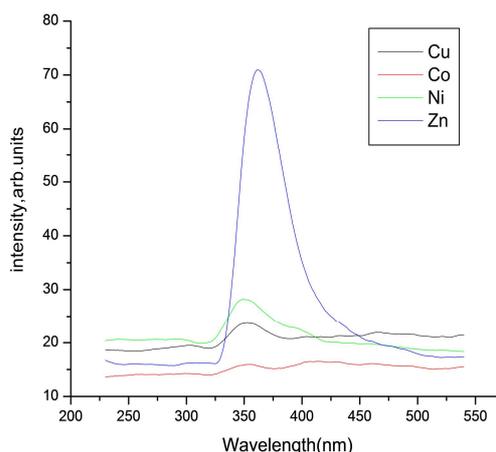
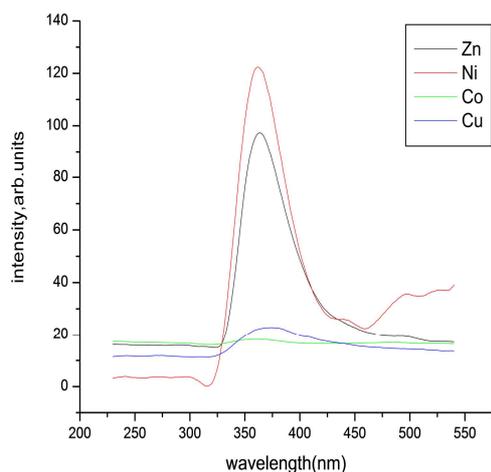


Figure 4. <sup>13</sup>C-NMR spectrum of Zn(II) complex

$\delta$ -149-150, C=C in the region of  $\delta$ - 137-140, C-N in the region of  $\delta$ -124.4-124.9. The L-1 metal complexes and L-2 metal complexes differ in the signals of C-Cl peak. Because the L-1 metal complexes do not get the peak of C-Cl, only the L-2 metal complexes show the signals of C-Cl in the region of 39.83 for Ni(II) and 41.42 only the L-2 metal complexes show the signals of C-Cl in the region of 39.83 for Ni(II) and 41.42 d 41.42 only the L-2 metal complexes show the signals of C-Cl in the region of 39. 83 for Ni(II) and 41.42 complexes

### Photoluminescence spectra data

Photoluminescence spectrum of metal complexes under the excitation of light at 220 nm is fig5 from the spectra it is understood that the transition metal complexes have the potential to emit light at specific wavelength. The L1 metal complexes of Ni, Zn, Cu, Co show emission peaks at 361 nm, 363nm, 375nm, and 370nm respectively. Similarly, the Photoluminescence spectra of the L2 metal complexes are shown in fig6. the emission peaks of the complexes are observed at 362nm, 351nm, 354nm, and 352nm for the metal Ni, Zn, Cu and Co respectively. These emission spectra indicate that the metal complexes of L2 have the ability to emit radiation at a different wavelength and the intensity of emission of light varies with respect to the nature of metals and ligand. In order to obtain more information about the application of the complexes as light emitting diode, Photoluminescence study to be carried out for the complexes in visible region. Further study on the electrochemical properties of the complexes will help explore the application of the complexes on light emitting devices based on energy band corresponding law.



**Fig 5: Photoluminescence spectra of the metal complexes (M-L1)**

**Figure 6. Photoluminescence spectra of the metal complexes (M-L2)**

#### IV MOLECULAR DOCKING STUDIES

The binding efficacy of the drug molecule and the protein is explained based on molecular docking technique. This technique plays pivotal role to understand the nature of binding between the receptor and the drug molecule. To predict the appropriate binding site along with preferable orientation of the molecule inside the protein, the free software gem dock is used .the experimental data indicates that the binding free energy of the molecule of L1 and L2 of zinc complexes with the protein is found to be significantly low(L1=-80.96,[Zn(L1)<sub>2</sub>(OH)<sub>2</sub>] =-90.29,L2=-71.9, [Zn(L1)<sub>2</sub>(OH)<sub>2</sub>] =-13.34).it is generally accepted that if the binding free energy is low, the potential of binding efficiency is more between the receptor and the drug molecule. Hence the docking studies of Isatin and it is derivatives of metal complexes reveals that zinc metal complexes with the ligand L1 and l2 can be used for drug designing against breast cancer as they are exhibiting excellent binding property with the BCAR1 protein.

Compounds	Energy	Vdw	H-Hond	Elec
[Cu(L1) <sub>2</sub> (OH) <sub>2</sub> ]	-90	-70.93	-19.4	0.32
[Co(L1) <sub>2</sub> (OH) <sub>2</sub> ]	-89.97	-70.77	-1.53	1.67
[Ni(L1) <sub>2</sub> (OH) <sub>2</sub> ]	-89.71	-70	-19.45	-0.26
[Zn(L1) <sub>2</sub> (OH) <sub>2</sub> ]	-90.29	-70.97	-19.32	0
[Cu(L2) <sub>2</sub> (OH) <sub>2</sub> ]	362.15	367.37	-9.8	4.58
[Co(L2) <sub>2</sub> (OH) <sub>2</sub> ]	228.19	228.05	-1.53	1.67
[Ni(L2) <sub>2</sub> (OH) <sub>2</sub> ]	363.21	328.993	4.23	0
[Zn(L2) <sub>2</sub> (OH) <sub>2</sub> ]	-13.34	-8.09	-5.25	0

#### V. CONCLUSION

Novel conjugated Isatin complexes of metal ions copper, cobalt, nickel and zinc are synthesized and characterized by UV-visible, IR and NMR spectral studies. The complexes showed distinct UV-visible absorption to bands and maximum emission bands at a specific wavelength with significant intensity. The photo luminescent study of the complexes indicated the metal complexes of novel conjugated Isatin ligands have the potential to exhibit the light emitting property and they should be expected to have good electron transporting and carrier balancing abilities that could pare away to make devices as OLED's.The biological property of the ligand and its metal complexes have been

# International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 8, August 2016

investigated by docking study. GEMDOCK online free software was used to find the binding efficacy of the ligand and metal complexes with BCAR1 protein expressed for breast cancer. The experimental study reveals that zinc complexes with L1 and L2 ligands have maximum potential against the breast cancer protein BCAR1.

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