SYNTHESIS, STRUCTURE AND PROPERTIES OF Zn(II) AND Cd(II) COMPLEXES WITH *(E)-N′*-(5-Br-2-HYDROXYBENZYLIDENE)-NICOTINOHYDRAZIDE (HL)

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Abstract: *New Zn(II) and Cd(II) complexes with a hydrazone based on (E)-N'-(5-bromo-2-hydroxybenzylidene)nicotinohydrazide (HL) were obtained. The structures of these complexes were studied using infrared (IR) spectroscopy, Electronic (UV–Vis) spectroscopy, derivatography, luminescence spectroscopy, and elemental analysis. It was established that Zn(II) forms a complex with the ligand (HL) in a metal-to-ligand ratio of M:L = 1:2, whereas Cd(II) forms a complex with a ratio of M:L = 1:1. Based on thermogravimetric analysis, the stages of thermal decomposition of the synthesized complexes and the composition of solid reaction products formed at these stages were determined. It was shown that the final products of thermal decomposition consist of zinc and cadmium oxides. The Zn(II) complex contains two water molecules, while the Cd(II) complex contains three water molecules. The complexes were dissolved in dioxane, and their luminescent properties were investigated. It was established that both the ligand and its complexes exhibit luminescent properties.*

Keywords: *Metal complexes, Schiff base, hydrazones, Zn(II), Cd(II) complexes, thermal analysis.*

Introduction

Hydrazide–hydrazone compounds represent a broad class of nitrogen-containing organic molecules formed as a result of condensation of acyl hydrazides with aldehydes or ketones. A common structural feature of these compounds is the presence of the functional group R–CO–NH–NH–CH=N–R′, which combines the properties of both amide and azomethine fragments. This structure provides hydrazide–hydrazones with an extended conjugation system, a variety of tautomeric forms, and a high ability for donor–acceptor interactions. As a result, they exhibit pronounced activity toward a wide range of transition and post-transition metal ions.

Interest in hydrazide–hydrazone ligands is due to the presence of several potential coordination centers— azomethine nitrogen, amide nitrogen, carbonyl oxygen, and, in the case of aromatic aldehydes, also phenolic oxygen.

These compounds are also of significant interest because of their biological activity. Hydrazide–hydrazone complexes have attracted considerable attention due to their antimicrobial [1–4], antimalarial [5], antioxidant [6–8], anticancer [9–13], antiviral [14, 15], and anti-inflammatory [16–18] activities.

Zinc and cadmium complexes with nitrogen-containing organic ligands are of interest due to their catalytic [19], biological [20, 21], and luminescent [22] properties. Compounds containing hydrazide–hydrazone ligands are particularly attractive because of their high coordination ability and antioxidant activity [23].

The aim of the present work is the synthesis and investigation of zinc and cadmium complexes with a hydrazide–hydrazone obtained by condensation of nicotinic acid hydrazide with 5-bromosalicylaldehyde, as well as the study of their structure and physicochemical properties.

Experimental Part

IR spectra were recorded on a Nicolet IS10 spectrophotometer in Vaseline oil in the range 4000–400 cm^{-1}. Electronic spectra were recorded on an Evolution-60S spectrophotometer. Thermogravimetric analysis was performed using a NETZSCH STA 449F3. Elemental analysis was carried out at the TÜBİTAK analytical laboratory (Ankara) using a LECO CHNS analyzer. Luminescent properties were measured on an Agilent Cary Eclipse fluorescence spectrophotometer.

1. Synthesis of Nicotinic Acid Hydrazide. Nicotinic acid hydrazide was synthesized according to the method described in [24].

M.p. = 159-160°C. Yield - 0.145 g (93.5%). IR (Vaseline oil) ν/cm^{-1}: 3321 w, 3190 w, 1651 w, 1596 m, 1571 w, 1463 s, 1377 s, 952 m, 721 m, 628 m, 524 m. UV/Vis (dioxane) λ_{max}, nm: 206, 262. Elemental analysis for $C_6H_7N_2O$ (123):

Calculated, %: C 58.54; H 5.69; N 22.76; O 13.01,

Found, %: C 58.51; H 5.71; N 22.74; O 13.05.

2. Preparation of N-nicotinoyl-N′-(5-bromo-2-hydroxy)benzylidene hydrazine (HL). A solution of 10^{-3} mol (0.137 g) of nicotinic acid hydrazide in 15 mL of ethanol was mixed with a solution of 10^{-3} mol (0.201 g) of 5-bromosalicylaldehyde in 20 mL of methanol. The mixture was heated at 60 °C for 30 min. Upon cooling to room temperature, light yellow crystals precipitated.

M.p. > 250°C. Yield: 0.28 g (83%) IR (in Vaseline oil) ν/cm^{-1}: 3659 w, 3159 s, 1624 w, 1562 w, 1475 s, 1283 w, 878 m, 823 w, 637 w. UV/Vis (dioxane) λ_{max}, nm; 244, 288, 300, 339. For $C_{13}H_{10}BrN_3O_2$ (320.15). Calculated, %: C 48.72; H 3.12; N 13.12; Br 24.96; O 10.00. Found, %: C 48.51, H 3.27, N 13.18, Br 24.66, O 10.05.

3. Synthesis of the Zn(II) Complex. A solution of 10^{-3} mol (0.32 g) of HL in 15 mL of ethanol was mixed with a solution of 10^{-3} mol (0.22 g) of zinc acetate dihydrate in 20 mL of methanol. The mixture was heated at 60°C for 30 min. Upon cooling, light yellow crystals formed. M.p.>250°C . Yield: 0.42 g (78%) IR (in Vaseline oil) ν/cm^{-1}: 3450 w, 1609 s, 1524 w, 1458 w, 1372 s, 1312 w, 1264 s, 1175 m, 1124 m, 966 m, 876 w, 824 w, 730 s, 623 w, 553 w, 494 m. UV/Vis (dioxane) λ_{max}, nm: 237, 294, 367.

For C26H22Br2¬N6O6 Zn (739.18)

Calculated, %: C 42.21; H 2.98; Br 21.62; N 11.36; O 12.99; Zn 8.84.

Found, %: C 42.18; H 2.85; N 11.34; Br 21.63; O 12.89; Zn 8.83.

4. Synthesis of the Cd(II) Complex. The Cd(II) complex was obtained by a similar procedure. M.p.>250°C. Yield: 0.32 g (89.6%) IR (in vaseline oil) ν/cm^{-1}: 3451 w, 1616 s, 1529 w, 1456 w, 1372 s, 1372 w, 1164 s, 946 m, 819 m, 725 m, 632 w, 480 w.

UV/Vis (dioxane) λ_{max}, nm: 357, 402, 425. For $C_{13}H_{13}BrN_3O_4Cd$ (485.31)

Calculated, %: C 32.14; H 3.09; Br 16.46; N 8.65; O 16.48; Cd 23.16.

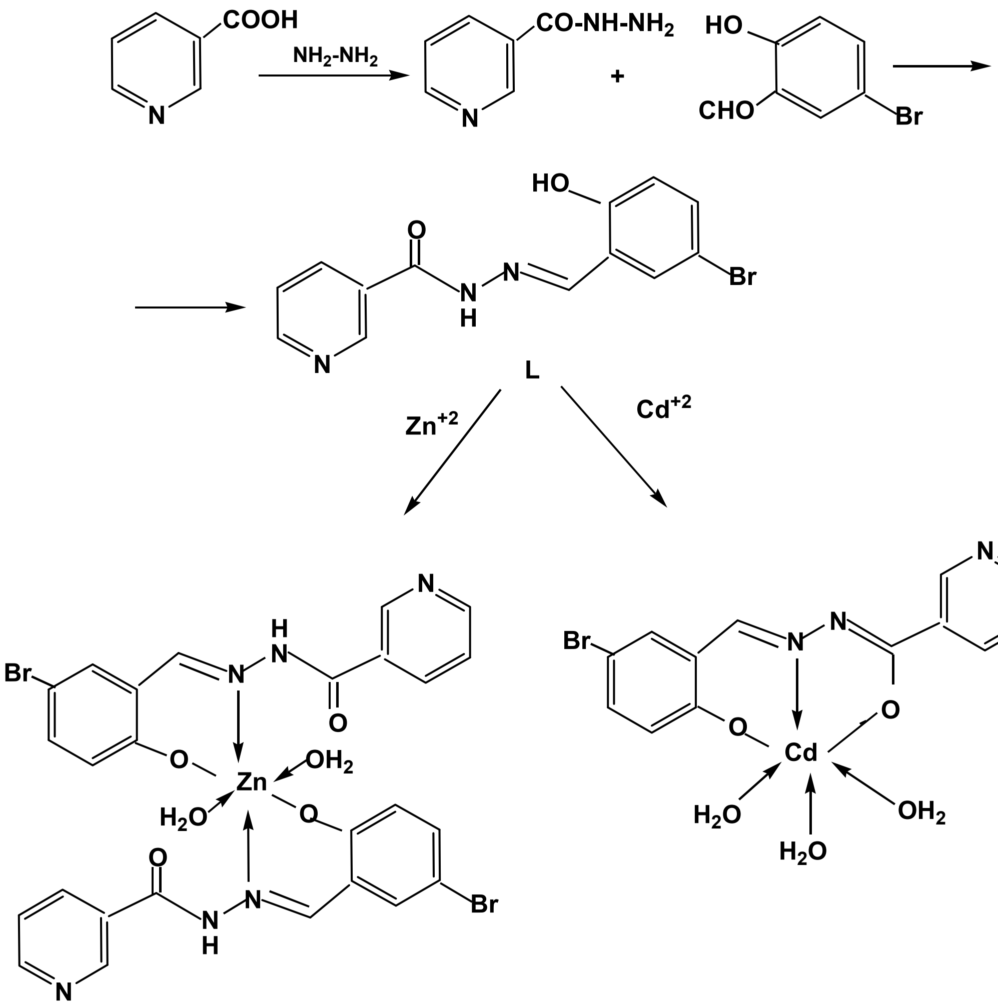
Found, %: C 32.12; H 3.12; Br 16.45; N 8.62; O 16.49; Cd 23.15.

The synthesis process is demonstrated in Scheme 1.

IR Spectra. In the IR spectrum of the Schiff base HL, the vibration of the sterically hindered phenolic OH group appears as a narrow band at 3619 cm^{-1}. The absorption band at 3159 cm^{-1} is attributed to NH vibrations of the amide group (NHCO). The C=N and C=O stretching vibrations are observed at 1624 and 1562 cm^{-1}, respectively. The band at 1475 cm^{-1} corresponds to aromatic ring vibrations [25]. Bands at 878 and 823 cm^{-1} are attributed to C–H vibrations of the pyridine ring [26].

In the IR spectra of the complexes, the amide NHC=O band at 1624 cm^{-1} disappears due to coordination with metal ions. Coordination of the azomethine group leads to a shift of the C=N band from 1624 cm^{-1} to 1609 cm^{-1} for the zinc complex and to 1616 cm^{-1} for the cadmium complex [26]. The ν(C–O) band of the phenolic group at 1283 cm^{-1} is significantly weakened due to deprotonation and participation of oxygen in coordination. New bands appear at 494 and 480 cm^{-1}, corresponding to Zn–O and Cd–O vibrations. In Cd complexes the shifts are less pronounced, but the ν(M-O) and ν(M-N) bands appear at lower frequencies (due to the heavier metal and longer Cd-O/N bonds).

The absence of the ν(O–H) band and the appearance of ν(M–O) and ν(M–N) bands confirm the involvement of phenolic groups in complex formation.



Scheme 1. Synthesis of the ligand (HL) and the complexes [Zn(2L)·2H2O] and [Cd(L)·3H2O]

Electronic Spectra. Since Zn(II) and Cd(II) ions have a d^{10} electronic configuration, d–d transitions are absent. The spectra are determined by intraligand (π–π* and n–π*) transitions and charge-transfer bands, predominantly ligand-to-metal charge transfer (LMCT). MLCTs are also possible in the presence of suitable low-energy π* orbitals of the ligand.

The UV–Vis spectra of the free hydrazone ligand and its Zn(II) and Cd(II) complexes were recorded in dioxane solution. The free ligand exhibits an intense π–π* band at 289 nm and a weaker n–π* band in the range 237–293 nm, which is characteristic of conjugated aromatic hydrazones. Upon coordination with Zn(II) and Cd(II), a long wavelength shift of the absorption bands was observed. For Zn(II), shifts of the π–π* and n–π* transitions by 7–10 nm toward longer wavelengths are characteristic, along with the appearance of a broad band at ~366–402 nm, which is interpreted as a ligand-to-metal charge transfer (LMCT) band. A shift toward longer wavelengths is also observed for the π–π* transitions. For Cd(II), the LMCT band is shifted further into the red region (~357–402 nm) and exhibits greater band broadening/intensity, which is consistent with the higher polarizability of Cd(II) and the lower energy of its acceptor orbitals. In the electronic spectrum of the Cd(II) complex, a weak LMCT band was observed at 425 nm (Fig. 1).

Changes in the positions and intensities of the bands upon going from the free ligand to the complexes confirm the involvement of the azomethine nitrogen and oxygen donors (phenolic O and C=O) in coordination, which is consistent with IR data and elemental analysis.

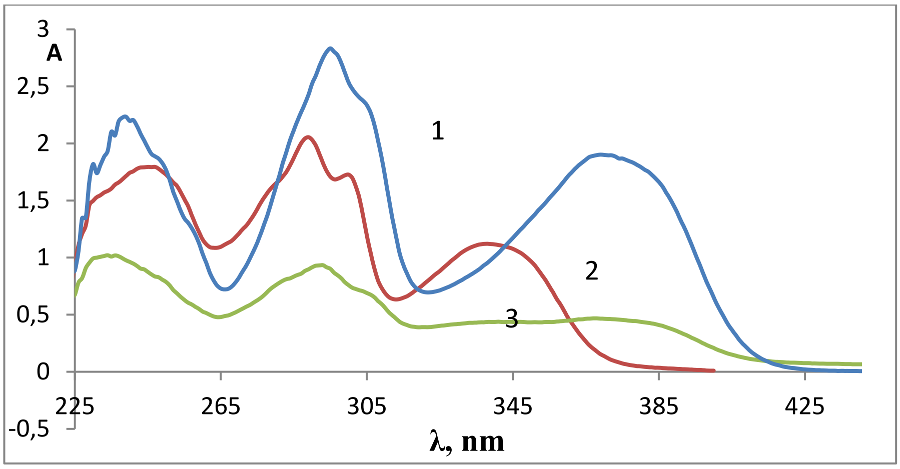


Fig. 1. Electronic absorption spectra of (1) the ligand, (2) the complex $[Zn(2L)\cdot 2H_2O]$, and (3) the complex $[Cd(L)\cdot 3H_2O]$ in dioxane

Derivatographic analysis. Derivatographic studies of the Zn and Cd complexes with the hydrazone of nicotinic acid and 5-bromosalicylaldehyde were carried out in the temperature range of 25–900°C at a heating rate of 10°C min^{-1} in a nitrogen atmosphere.

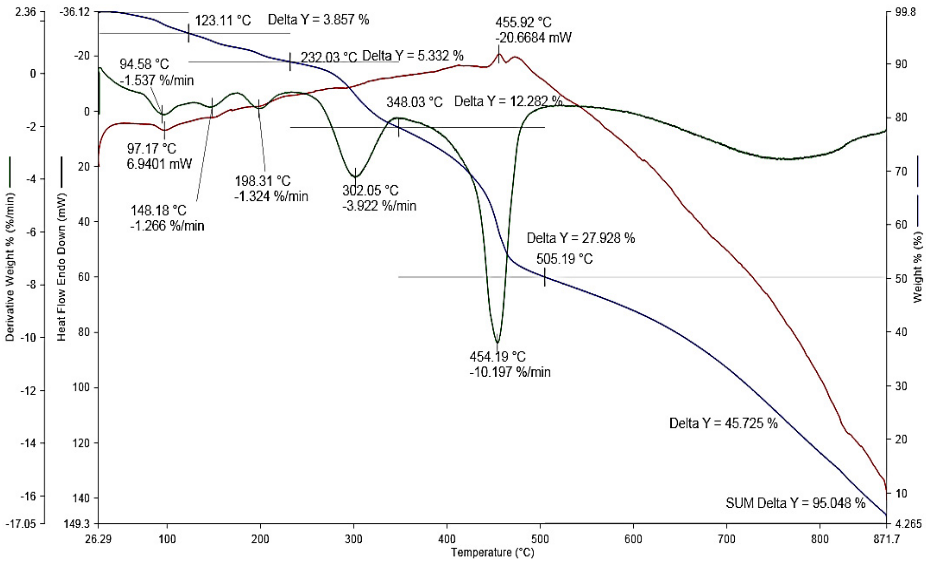


Fig. 2. Derivatogram of the complex $[Zn(2L)\cdot 2H_2O]$

On the thermogravimetric (TG) curves of both complexes, three main stages of mass change are observed. The first stage (30–150 °C) corresponds to the removal of crystallization water or solvent (mass loss of 3.9% for $[Zn(2L)\cdot 2H_2O]$ and 4.9% for $[Cd(L)\cdot 3H_2O]$), which is consistent with the expected presence of two crystallization water molecules in the Zn(II) crystal lattice and three water molecules in the Cd(II) crystal lattice. The second stage (140–350 °C) is associated with partial degradation of the organic fragment of the ligand, involving the cleavage of peripheral aromatic groups (mass loss of 5.3–12.3%) (Fig. 2).

The main stage of thermal decomposition is observed in the range 290–560 °C (T_{max} = 505 °C for the Zn complex and T_{max} = 588 °C for the Cd complex) and is accompanied by an intense mass loss (34–45%), reflecting complete destruction of the organic part of the ligand and formation of a thermally stable inorganic residue. The residual mass at 700–900 °C is 45,2–43%, which is in good agreement with the theoretically calculated values for ZnO and CdO, respectively. The DTA curves show pronounced extrema in the region of the main stage: exothermic peaks when measurements are carried out in air (corresponding to oxidation of the organic matter) and smoother changes when measurements are performed in an N_2 atmosphere (Fig. 3).

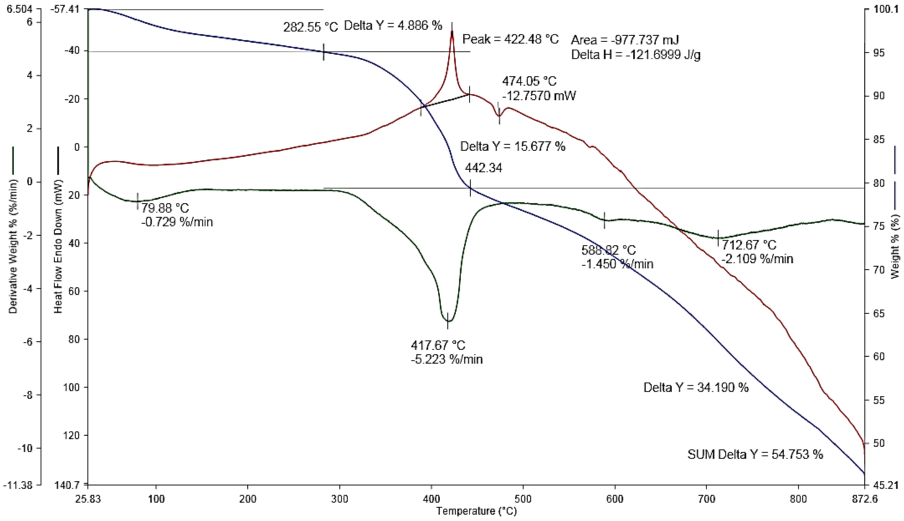


Fig. 3. Derivatogram of the complex [Cd(L)·$3H_2O$]

Thus, the derivatographic data confirm that the studied complexes undergo thermal decomposition with the formation of the corresponding metal oxides (ZnO and CdO), and the differences in the temperature maximums and decomposition profiles reflect differences in the polarizability and coordination ability of Zn^{2+} and Cd^{2+} ions.

Luminescent properties. Zn(II) and Cd(II) ions do not exhibit d–d transitions; therefore, they do not quench luminescence. On the contrary, they stabilize the excited state of the ligand and suppress non-radiative relaxation pathways. The emission originates mainly from intraligand transitions (π–π*, n–π*) and charge-transfer transitions (LMCT, ILCT).

Coordination to a metal ion usually results in a shift of the emission band toward longer wavelengths (red-shift).

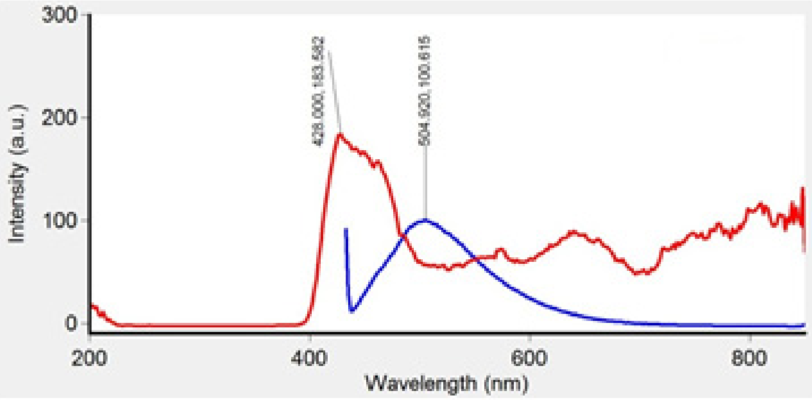


Fig. 4. Luminescence of the ligand (E)-N′-(5-Br-2-hydroxybenzylidene)nicotinoyl hydrazone (HL) in DMF

The luminescent properties of the hydrazone ligand and its Zn(II) and Cd(II) complexes were investigated in solution upon excitation in the 400–550 nm region. The free ligand exhibits an emission band at 505 nm, which is attributed to intraligand π–π* and n–π* transitions (Fig. 4).

In the spectra of the complexes, a shift of the emission bands toward longer wavelengths and a significant increase in emission intensity are observed, indicating effective suppression of vibrational relaxation as a result of ligand coordination to d^{10} metal ions. The Zn(II) complex shows bright blue emission with a maximum at 540 nm (Fig. 5), whereas the Cd(II) complex is characterized by broader blue-green emission (λ_{em} = 481 nm) (Fig. 6). The increase in the Stokes shift and the differences in band positions reflect the different polarizabilities of Zn^{2+} and Cd^{2+} ions, as well as differences in electron density distribution in the excited state.

The absence of d–d transitions and the presence of pronounced ILCT (intraligand charge-transfer) transitions make these compounds promising candidates for luminescent sensors and optoelectronic materials.

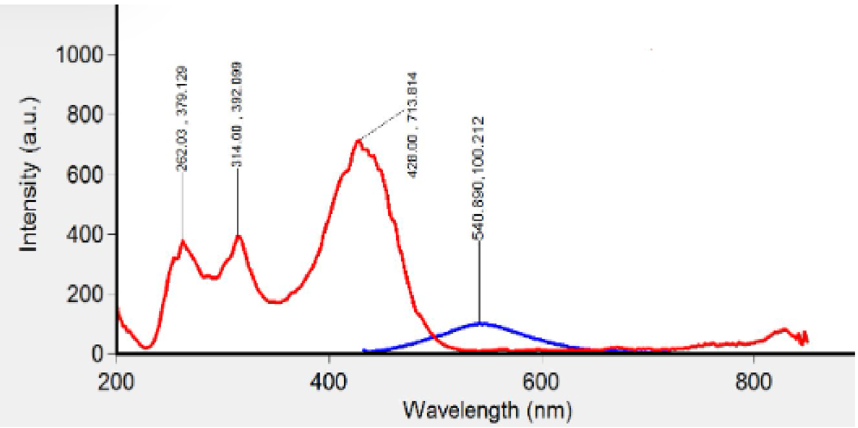


Fig. 5. Luminescence of the complex [Zn(2L)·$2H_2O$]

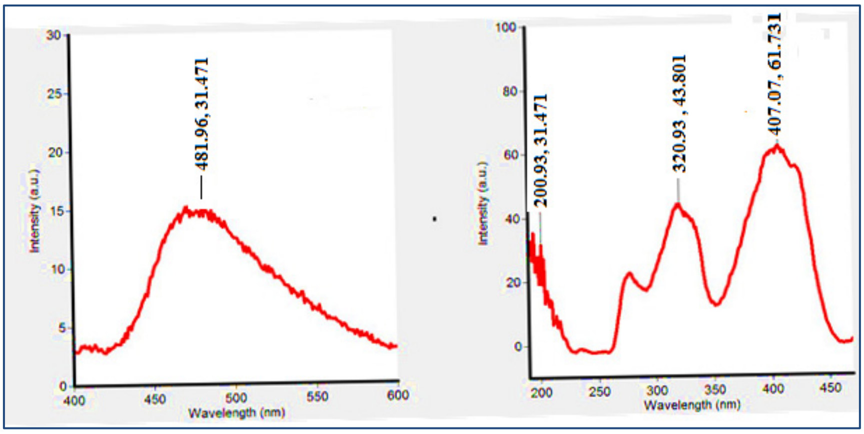


Fig. 6. (a) Luminescent and (b) electronic spectra of the complex [Cd(L)·$3H_2O$]

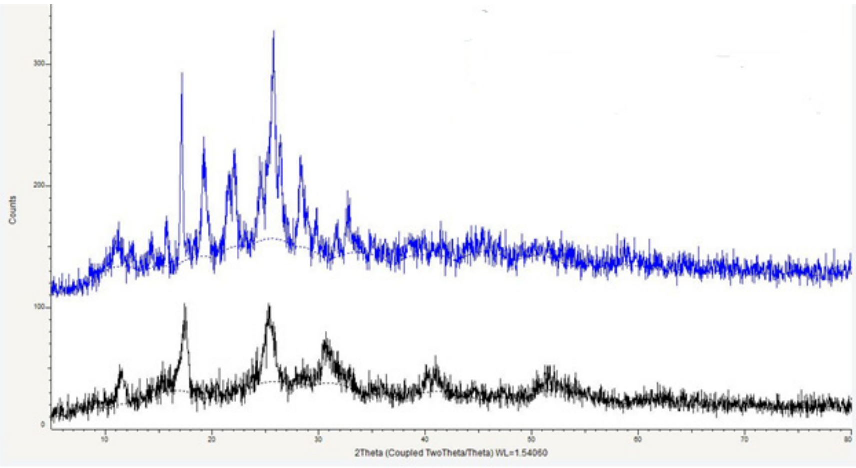


Fig. 7. X-ray diffraction patterns of the combustion products of (1) [Zn(2L)·$2H_2O$] and (2) [Cd(L)·$3H_2O$].

X-ray diffractometric data showed that the complexes [Zn(2L)·2H_2O] and [Cd(L)·3H_2O] are not isostructural (Fig. 7).

Conclusion

New Zn(II) and Cd(II) complexes with (E)-N′-(5-bromo-2-hydroxybenzylidene) nicotinoyl hydrazone (HL) were synthesized and characterized. The ligand forms complexes with metal ions of different compositions. In the zinc complex [Zn(2L)·2H_2O], the ligand is coordinated to the metal ion through the oxygen atoms of two phenolic groups and the azomethine nitrogen atom. In the cadmium complex [Cd(L)·3H_2O], the ligand is coordinated to the metal ion through the phenolate oxygen and the carbonyl oxygen in the enol form, as well as the azomethine nitrogen atom. It was found that all the obtained compounds exhibit luminescent properties.

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