

Research Article

Thermal, Electrical, and Optical Properties of Synthesized (1*E*, 2*E*)-(4-bromophenyl)(hydroxyimino)acetaldehyde Oxime Complexes

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The (1*E*, 2*E*)-(4-bromophenyl)(hydroxyimino)acetaldehyde oxime complexes, [ML₂], M = Co(II), Cu(II), and Ni(II), were synthesized and characterized by elemental analysis, magnetic susceptibility, FT-IR spectra, and thermal analysis techniques. The optical band gap of this ligand and its complexes were determined by UV-vis spectrophotometer in the wavelength range 300–800 nm. A decrease in the energy band gap of the [CoL₂], [NiL₂], and [CuL₂] complexes has been observed compared with LH ligand. Temperature-dependent conductivity measurements showed that all samples behave like semiconductor. Activation energies calculated from Arrhenius plots showed that the conduction occurs via both intramolecular and the intermolecular processes.

1. Introduction

Oximes represent a very significant group of ligands in coordination chemistry [1–5]. Dioximes, together with their complexes, are of interest for many researchers in different fields of chemistry. They have enormous importance in coordination chemistry, as many complexes of dioximes with transition metal ions have been isolated and characterized [6, 7].

The exceptional stability and unique electronic properties of the complexes can be attributed to their planar and tetrahedral structure, which is stabilized by hydrogen bonding [8, 9]. The high stability of the complexes prepared with oxime ligands has been used extensively for various purposes. The semiconductor properties of the complexes synthesized by Schrauzer and Windgassen have been reported [8, 10]. Inorganic semiconducting complexes constitute one of the most fascinating recent research topics, deeply involving both

chemists and solid state physicists [8, 11]. Considerable interest has been shown in the synthesis and study of molecular complexes, which may behave like semiconducting materials [8, 12]. Semiconducting organic solid materials are frequently grouped into the categories of molecular crystals, charge transfer complexes, and polymers. Inorganic semiconductors stand on the threshold of a bright and exciting future. An organic semiconductor can be synthesized with properties comparable to those exhibited by inorganic semiconductor materials such as development for transistors and the wide array of now-existing derivative devices and components of the electronics industry [13].

The purpose of this study is to investigate some chemical and physical properties of newly synthesized metal complexes. The effects of Co, Ni, and Cu to Ligand on the thermal, electrical, and optical properties have been investigated by means of thermal analysis, optical absorption, and temperature-dependent conductivity measurements.

2. Experimental

All remaining reagents were purchased from Merck or Fluka or Sigma Company and were used without further purification. Elemental analyses (C, H, and N) were determined using a Leco, CHNS-932 model analyzer. ^1H NMR spectra were recorded on a Bruker DPX 400 MHz High Performance Digital FT-NMR and IR spectra on a Perkin Elmer 1605 FTIR Spectrometer in KBr pellets. Magnetic susceptibilities were determined on a Sherwood Scientific Magnetic Susceptibility balance (Model MK1) at room temperature using $\text{Hg}[\text{Co}(\text{SCN})_4]$ as a calibrate; diamagnetic corrections were calculated from Pascal's constants. The metal analyses were determined using a PerkinElmer Model Analyst 700 flame atomic absorption spectrometer. Shimadzu DTG-60H thermal analyzer was used to record simultaneous TG, DTG, and DTA curves in a dynamic N_2 atmosphere with a $50\text{ cm}^3\text{ min}^{-1}$ flow rate, at a heating rate of 10 K in the $20\text{--}900^\circ\text{C min}^{-1}$ range using platinum crucibles. Highly sintered $\alpha\text{-Al}_2\text{O}_3$ was used as a reference, and the DTG sensitivity was 0.05 mg s^{-1} . Optical absorption spectra were taken by using a UV-vis absorption spectrophotometer (Shimadzu 160A Double Beam), in the wavelength range 190–1100 nm.

For the electrical measurements, typical sample dimension and thickness were $5 \times 5\text{ mm}^2$ and $100\text{ }\mu\text{m}$, respectively. For the electrical measurements the electrodes were attached to the contact regions with silver paint. The ohmic behaviour of the contacts in the studied temperature region was confirmed by the linear variations of the I - V characteristics, which are independent of the reversal of the applied currents. The temperature-dependent conductivity measurements in the temperature range of 260–450 K were carried out by using a Keithley 2400 source measure unit. The samples were placed onto cold finger of a Janis liquid nitrogen cryostat, and the temperature was accurately monitored with a Lake-Shore 320 temperature controller.

The preparation of isonitroso-*p*-bromoacetophenone has been described previously [14–17].

2.1. Synthesis of (1E, 2E)-(4-bromophenyl)(hydroxyimino)acetaldehyde Oxime (LH). LH ligand has already been synthesized as reported in the literature by [18, 19]. *P*-Bromophenylglyoxime [(1E, 2E)-(4-bromophenyl)(hydroxyimino)acetaldehyde oxime] was resynthesized from isonitroso-*p*-bromoacetophenone similarly according to previously published methods [17, 20]. The structure of the ligand is shown in Figure 1.

A quantity of 0.05 mol (11.4025 g) isonitroso-*p*-bromoacetophenone was dissolved in 50 mL ethanol. Subsequently, solutions of 0.06 mol (4.1694 g) $\text{NH}_2\text{OH}\cdot\text{HCl}$ and 0.18 mol (24.4944 g) $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$ (dissolved in the minimum amount of water) were added with stirring. The reaction mixture was refluxed for 5 h, and then excess ethanol in the reaction mixture was removed in vacuo. The precipitate was filtered and then recrystallized in ethanol-water (1:2) mixture and dried at 60°C . Colour: Colourless, Yield: 8.14 g (%67), M.p.: 159°C , Anal. Calcd (Found) for $\text{C}_8\text{H}_7\text{BrN}_2\text{O}_2$: C: 39.53(39.61), H: 2.90(2.29), N: 11.53(11.23),

IR (cm^{-1}): $\nu_{(\text{O}-\text{H})}$: 3232 m, $\nu_{(\text{C}-\text{H})(\text{arom})}$: 3019 m, $\nu_{(\text{C}-\text{H})(\text{aliph})}$: 2880 w, $\nu_{(\text{C}=\text{N})}$: 1668 s, $\nu_{(\text{N}-\text{O})}$: 999, ^1H NMR DMSO- d_6 δ (p.p.m.): O–H: 12.06 s (1H), O–H: 11.96 s (1H), H_{Arom} : 7.23–7.84 m (4H), $-\text{CH}_{\text{Alip}}$: 8.37 s (1H).

2.2. Ni(II), Co(II), and Cu(II) Complexes of Ligand. A solution of 2 mmol metal salt [$\text{NiCl}_2\cdot 6\text{H}_2\text{O}$ (0.4756 g), $\text{CoCl}_2\cdot 6\text{H}_2\text{O}$ (0.4760 g), $\text{CuCl}_2\cdot 2\text{H}_2\text{O}$ (0.3408 g)] dissolved in EtOH (30 cm^3) was added to a stirred solution of the ligand (0.9722 g, 4 mmol) dissolved in absolute EtOH (30 cm^3). On addition of the metal salt, the pH dropped to 3.5–4.0 from 5.0–5.5 at the onset of the reaction. After addition of a 1% KOH solution in EtOH to raise the pH to the mixture was stirred on a water bath at $55\text{--}60^\circ\text{C}$ for 1 h. The precipitated complexes were filtered off, washed with H_2O , and dried in vacuum at 60°C .

Ni(II) complexes [NiL_2]: Colour: Tile red, Yield: %73, M.p.: 315°C (dec.), μ_{eff} (B.M.): Dia., Anal. Calcd (Found) for $\text{C}_{16}\text{H}_{12}\text{Br}_2\text{N}_4\text{O}_4\text{Ni}$: C: 35.40(35.33), H: 2.23(2.25), N: 10.32(10.31), Ni: 10.81(10.66), IR (cm^{-1}): $\nu_{(\text{O}-\text{H})}$: 3400 m, $\nu_{(\text{C}-\text{H})(\text{arom})}$: 3062 m, $\nu_{(\text{C}-\text{H})(\text{aliph})}$: 2922 w, $\nu_{(\text{C}=\text{N})}$: 1653 w, $\nu_{(\text{N}-\text{O})}$: 1008 m.

Co(II) complexes [CoL_2]: Colour: Brown, Yield: %69, M.p.: 279°C (dec.), μ_{eff} (B.M.): 2.20, Anal. Calcd (Found) for $\text{C}_{16}\text{H}_{12}\text{Br}_2\text{N}_4\text{O}_4\text{Co}$: C: 35.38(35.41), H: 2.23(2.18), N: 10.32(10.17), Co: 10.85(10.82), IR (cm^{-1}): $\nu_{(\text{O}-\text{H})}$: 3369 m, $\nu_{(\text{C}-\text{H})(\text{arom})}$: 3085 m, $\nu_{(\text{C}-\text{H})(\text{aliph})}$: 2970 w, $\nu_{(\text{C}=\text{N})}$: 1650 w, $\nu_{(\text{N}-\text{O})}$: 1009 m.

Cu(II) complexes [CuL_2]: Colour: Dark green, Yield: %90, M.p.: 324°C (dec.), μ_{eff} (B.M.): 1.72, Anal. Calcd (Found) for $\text{C}_{16}\text{H}_{12}\text{Br}_2\text{N}_4\text{O}_4\text{Cu}$: C: 35.09(34.89), H: 2.21(2.09), N: 10.23(10.04), Cu: 11.60(11.71), IR (cm^{-1}): $\nu_{(\text{O}-\text{H})}$: 3386 m, $\nu_{(\text{C}-\text{H})(\text{arom})}$: 3060 m, $\nu_{(\text{C}-\text{H})(\text{aliph})}$: 2970 w, $\nu_{(\text{C}=\text{N})}$: 1626 w, $\nu_{(\text{N}-\text{O})}$: 1009 m. TGA data of the complexes and electronic parameters are given in Table 1.

3. Results and Discussion

The Cu(II), Ni(II), and Co(II) complexes of the (1E, 2E)-(4-bromophenyl)(hydroxyimino)acetaldehyde oxime were obtained in ethanol by the addition of sufficient 1% KOH in EtOH to increase the pH to 5.0–5.5. In all of these complexes, the metal/ligand ratio determined by the elemental analysis was found to be 1:2 as found for most of the oximes [20–24]. The structures of the complexes are shown in Figure 2. Since the synthesized metal complexes were not sufficiently soluble in any solvents, we were not able to study their ^1H NMR properties. Thus, the spectral studies are limited only to the IR spectroscopy, thermal analysis, magnetic susceptibilities, and elemental analyses. These measurements were considered to provide sufficient evidence to describe the structure of the metal complexes.

In the IR spectrum of the ligand, the O–H stretching vibrations were observed at 3232 cm^{-1} . In the IR spectrum of the paramagnetic metal complexes, the –OH stretching band appears at ca. 3400, 3369, 3386 cm^{-1} indicating that free –OH groups are present in the molecule. The stretching vibrations belonging to C=N and N–O groups are observed

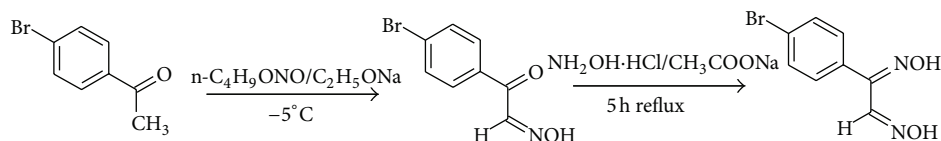


FIGURE 1: Schema of the synthesis of the ligand.

TABLE I: TGA data and electronic parameters of ligand and its complexes.

Compounds	Stability	Thermal properties			Residue	Electronic parameters			
		I. Step	II. Step	Weight loss % (calcd/found)		E_{gD} (eV)	E_1 (eV)	E_2 (eV)	E_3 (eV)
LH	20–121	121–390	—	100.0 (99.96)	—	4.20	333	—	—
[NiL ₂]	20–196	196–330	330–502	86.24 (85.87)	NiO	3.95	131	401	—
[CoL ₂]	20–217	217–298	364–422	86.24 (85.93)	CoO	3.55	113	375	137
[CuL ₂]	20–143	172–224	365–573	88.40 (87.46)	CuO	3.95	214	337	92

E_a : activation energy of electrical conduction, E_{gD} : energy gap for allowed direct transitions.

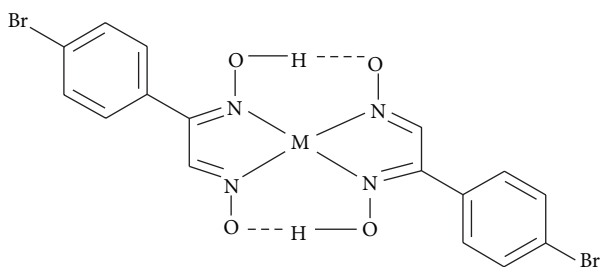


FIGURE 2: Metal complexes of the ligand [M = Ni(II), Cu(II), and Co(II)].

at 1668 and 999 cm^{-1} , respectively. These values are in harmony with the previously reported oxime derivatives [14–16, 25, 26]. In the IR spectrum of the mononuclear Ni^{2+} , Co^{2+} , and Cu^{2+} complexes, the infrared band observed near 1668 cm^{-1} assigned to the C=N frequency in the free ligand is shifted to lower frequencies (1653, 1650, and 1626 cm^{-1}) after complexation due to N,O-metal coordination for the Ni^{2+} , Co^{2+} , and Cu^{2+} complexes [27, 28]. At the same time, the band observed at 999 cm^{-1} in the free ligand, which was assigned to N–O, is shifted to higher frequencies (1008 and 1009 cm^{-1}) after complexation [29, 30].

Magnetic susceptibility measurements provide valuable information on the structure of complexes. The magnetic moments of the complexes were measured at room temperature and reported in the experimental section. The magnetic moments of the Ni(II), Co(II), and Cu(II) complexes of the ligand at room temperature fall in the range 0 μB , 2.20 μB , and 1.72 μB for Ni(II), Co(II), and Cu(II), respectively. The mononuclear complex of Ni(II) is diamagnetic, while Co(II) and Cu(II) are paramagnetic. According to the literature, square-planer geometry of the structure of the Ni(II), Co(II), and Cu(II) complexes may be reasonable [29, 31–33].

The decomposition temperatures and the weight losses of all the complexes were calculated from the t.g.a. measurements. While ligand decomposing one step, complexes

decomposed in two steps at different temperature ranges. In the t.g.a. curve of the complexes, weight loss was not observed at ca. 130–140°C. This shows that the complexes contain no water. It is known that the electronegativity and the atomic radius of the central metal atom also affect the thermal stability [34]. The inflation of the t.g.a. curves of all the complexes at a temperature below 800°C indicates the decomposition of the fully organic part of the chelate, leaving the metallic oxide at the final temperature [33, 35, 36].

In order to examine the effect of Co, Cu, and Ni bonded to the synthesized LH ligand, $[\text{ML}_2]$, M = Co(II), Cu(II), and Ni(II), optical absorption measurements have been carried out at room temperature. In Figures 3(a)–3(d), the spectra of the samples are presented in the spectral range 300–800 nm. In general, the absorption coefficient of the semiconductors obeys the equation

$$\alpha h\nu = A(h\nu - E_g)^n, \quad (1)$$

where A is a constant, E_g is the band gap, and n is a parameter which can be assumed to have values of 1/2 and 2 depending on the nature of electronic transition responsible for the absorption: $n = 1/2$ for a direct-allowed transition and $n = 2$ for an indirect-allowed transition. Therefore, the dependence of $(\alpha h\nu)^n$ on photon energy ($h\nu$) was plotted of $n = 2$ for direct transitions in Figures 3(a), 3(b), 3(c), and 3(d). Hence a straight line graph can be plotted between $(\alpha h\nu)^2$ and $h\nu$. The intercept to the $h\nu$ axis gives the direct energy band gap of the samples. It was found that all the investigated complexes have direct band gap due to direct transitions. The optical band gaps have been determined and given in Table 1. It is seen from Figure 3 that there is a shift in the absorption peak of the $[\text{CoL}_2]$, $[\text{NiL}_2]$, and $[\text{CuL}_2]$ complexes compared with LH. A decrease in the energy band gap towards lower photon energies was found. The decrease in the energy band gap gives rise to the increase in dc conductivity of the complexes. As can be seen in Figure 3(b), the dominant decrease in the optical band gap has been observed for the sample $[\text{ML}_2]$, M = Co(II) with a broadening of the absorption edge. Such a band gap narrowing and

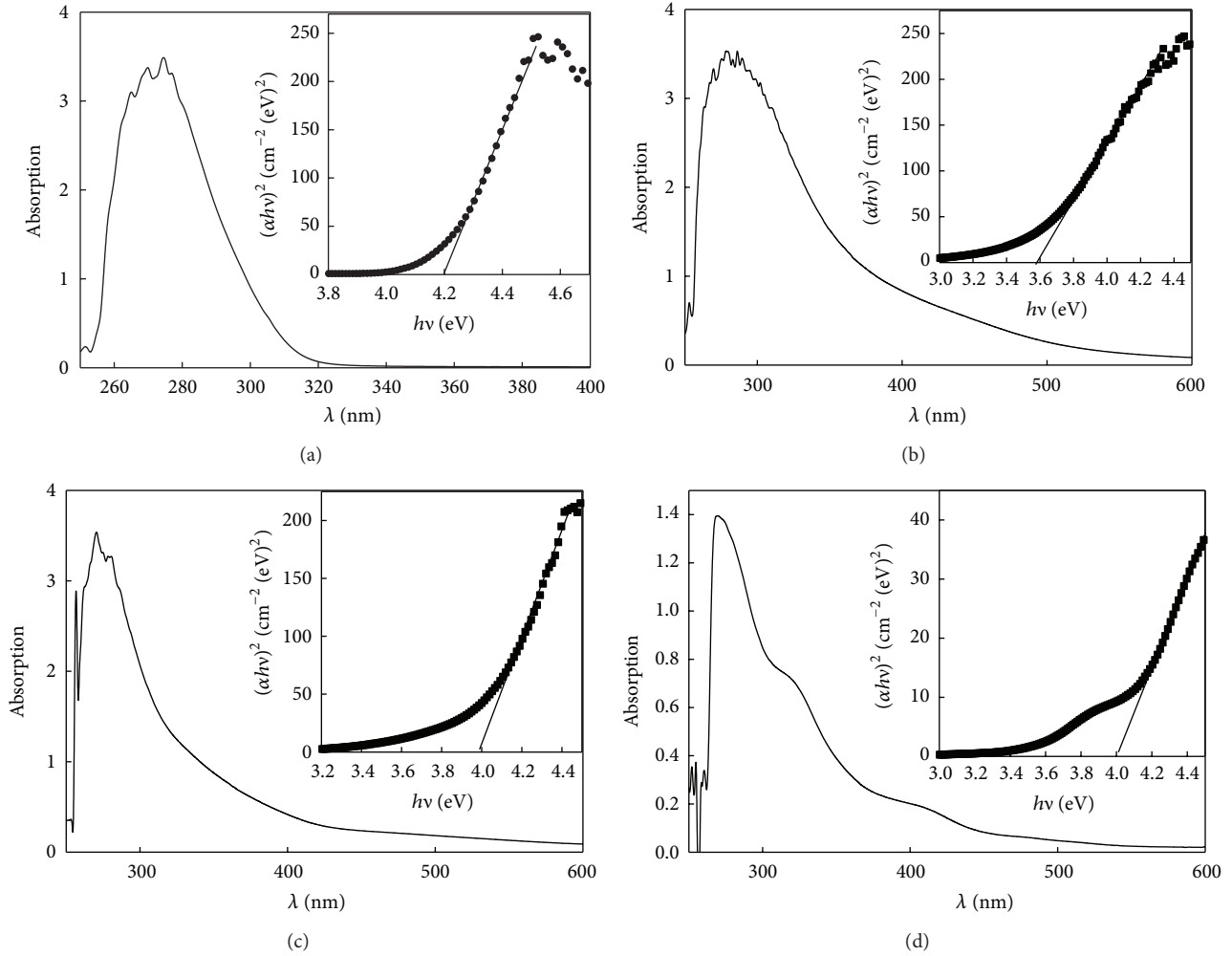


FIGURE 3: Absorption spectra of (a) synthesized ligand LH and its complexes (b) L_2Co , (c) L_2Cu , and (d) L_2Ni at room temperature.

band tails were reported in the absorption spectra of CdSe thin films depending on increasing in concentration [37, 38]. This behavior was explained with the structural disorder induced by the lattice mismatch and the increase of the free carriers with doping density which reduces the intensity of the oscillator strength of the discrete exciton states. Therefore, the absorption edge of the sample $[ML_2]$, $M = Co(II)$ is characterized by continuum state transitions.

Temperature-dependent conductivities of the LH, $[CoL_2]$, $[NiL_2]$, and $[CuL_2]$ in the temperature range of 260–450 K are shown in Figure 4. The electrical conductivity of the LH ligand and its complexes have positive temperature coefficient. That is, the conductivity of the all samples is increased exponentially with increase of temperature in the studied temperature range, indications of the semiconducting nature. It is seen from Figure 4 that the bounding of Co, Cu, and Ni to the LH ligand causes an increase in conductivity. Similar to the absorption measurements, in which the dominant decrease in the optical band gap has been recorded for the sample $[CoL_2]$, the highest decrease in resistivity has been observed for the sample $[ML_2]$, $M = Co(II)$. While the room temperature resistivity of the L was found to

be $1.7 \times 10^{10} \Omega\text{-cm}$, the resistivities at room temperature for $[CoL_2]$, $[NiL_2]$, and $[CuL_2]$ were reduced down to the values 1.6×10^9 , 8.4×10^9 , and $6.9 \times 10^9 \Omega\text{-cm}$, respectively. The temperature-dependent conductivities in the whole temperature range were analyzed by using the conductivity expression

$$\sigma = \sigma_o \exp\left(-\frac{E_a}{kT}\right), \quad (2)$$

where σ_o is the preexponential factor, E_a is the activation energy for thermally activated process, and k is the Boltzmann constant. According to (2), the activation energies for different temperature intervals can be found from the slope of linear regions of $\ln \sigma - 1000/T$ plot. Activation energies calculated for all samples are given in Table 1, for different temperature intervals. It is seen from Figure 4 and Table 1 that while LH has only one activation energy in the whole investigated temperature range, $[NiL_2]$ has two activation energies and $[CoL_2]$ and $[CuL_2]$ have three activation energies. These different activation energies are associated with the intramolecular and the intermolecular conductivity processes. In general, while the lower values

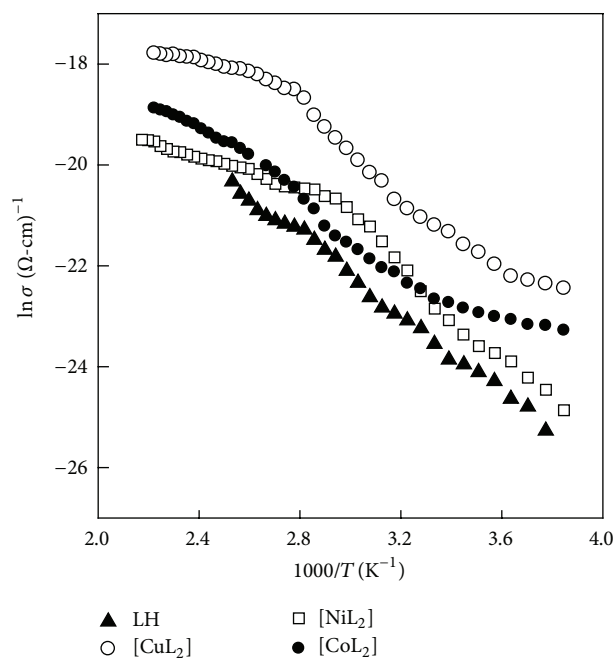


FIGURE 4: Temperature dependence of the electrical conductivity of synthesized ligand and its complexes.

of E_a are associated with the intermolecular conduction process, the higher values are related with the intramolecular conduction process [39]. Therefore, we may conclude that transport processes take place via two ways in different temperature range and the metal complexes introduce local defects, in which orbital overlap and electrons or holes can travel from one kind of macromolecule to another.

4. Conclusions

The (1*E*, 2*E*)-(4-bromophenyl)(hydroxyimino)acetaldehyde oxime complexes, [ML₂], M = Co(II), Cu(II), and Ni(II) were obtained. The structure was square-planer geometry. Spectroscopic measurements showed that metal complexes make the physicochemical properties more stable. Absorption measurements showed that there is a shifting in the absorption peak after the bounding of the metal complexes to the LH ligand and the optical band gap values are decreasing for [CoL₂], [NiL₂], and [CuL₂] complexes compared with LH. The decrease in the energy band gap gives rise to the increase in dc conductivity of the complexes. Transport processes take place via intramolecular and the intermolecular conductivity processes in different temperature range, and the metal complexes introduce local defects.

Disclosure

All the authors declared that they have no financial relationships—Merck, Fluka, Sigma or any other companies.

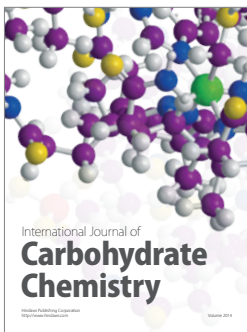
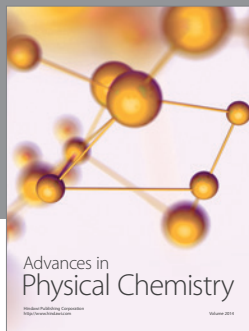
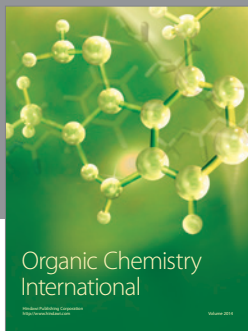
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