Commun.Fac.Sci.Univ.Ank.Series B
Volume 60, Number 2, Pages 1-16(2018)
DOI: 10.1501/commub_0000000558
ISSN 1303-6017
http://communications.science.ankara.edu.tr/index.php?series=B

COORDINATION POLYMERS BASED ON MIXED CARBOXYLATE LIGANDS: SYNTHESIS AND THERMAL STUDIES

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Abstract. The synthesis of Co(II), Cu(II), Fe(II) and Zn(II) based coordination compounds using terephthalic acid (BDC), tartaric acid (DHB), and 4-hydroxybenzoic acid (4-HBA) as ligands by a solvent-based and solvent-free methods were reported. Thermal studies of the complexes performed in the temperature range of 30 ŋC-950 ˚C showed a 9.406% weight loss observed between 200 ŋC-517 ŋC for [Co(DHB)(4-HBA)] complex, and a 1.883 % weight loss observed between 80 ŋC-352.68 ŋC for [Cu(DHB)(4-HBA)] complex.

1. Introduction

Porous materials generally are solids which have pores in their frameworks which act as spaces that can accommodate other guest molecules [1]. Organic carbon (i.e. activated carbon) and zeolites (inorganic frameworks) are the commonly known porous materials. Activated carbon is known to have high surface areas and high adsorption capacities; however, it lacks an ordered structure. Despite this limitation, porous carbon materials have found applications in various processes which include storage of gases, shape and size-selective catalysis, and templates for low dimensional materials [2,3].

Inorganic porous materials (e.g. zeolites) usually exhibit ordered frameworks and their synthesis often entails the use of inorganic or organic templates with strong interaction existing between the inorganic moiety and the template. There is a lack of diversity in the earlier known inorganic frameworks due to the elements used being limited mostly to either Al or Si. Removal of the template sometimes results in the breakdown of the framework. These frameworks, however, have found applications in catalysis and molecular separations [1,3].

Received by the editors: September 09, 2018; Accepted: December 15, 2018.
Key word and phrases: Metal complexes, Solvent-based method, Terephthalic acid, Thermal studies

Communications Faculty of Sciences University of Ankara Series B: Chemistry and Chemical Engineering

Porous hybrid materials can thus be prepared which incorporates the properties of both organic and inorganic porous materials. These hybrid solids have the advantage of being stable, possessing highly ordered structure and high surface area. Application of these hybrid solids has spanned drug storage and delivery, gas storage/separation, size-, shape-, and enantio-selective catalysis [4,5].

Synthetic chemistry has been an area of chemistry which has led to the production of solids with interesting properties such as high porosity. Solids which contain metal ions that are linked together by molecular species have featured mostly in the study of materials which are known to be porous and which has been exploited for various applications [6-8]. Metal-organic frameworks (MOFs) are essentially a class of hybrid porous materials also known as coordination polymers/networks prepared by the linking together of metal ions with organic linkers (ligands) leading to the formation of interesting structural topologies [9]. The polyfunctional ligands form coordinate bonds with multiple metal atoms resulting in one-, two-, or three-dimensional extended polymeric structures [10].

Research interest has increased in the synthesis of coordination polymers/networks because of their potential usage in catalysis, molecular separation, luminescence, gas storage, and sorption. The extent to which a coordination polymer is robust at high temperature and its structural stability in the presence of various organic, inorganic and adverse conditions determines its effectiveness [11].

A variation of the ligands and metal ions with the synthesis conditions has led to the preparation of a variety of compounds having several interesting properties and a series of application [12-14]. The ligand character (i.e. ligand length, chirality, bond angles, and bulkiness) influences the resultant framework. The inclination of the metal ion to take on specific geometries also contributes to the type of structure the coordination compound will attain [1,15-17].

Carboxylic acid ligands have been known to adopt versatile coordination modes by the bridging of different metal centres resulting in a variety of thermally stable coordination networks/polymers. Some coordination compounds have been constructed by the π-π stacking and hydrogen bond formation between polymer chains [18]. Aromatic carboxylate ligands have the ability to achieve a porosity of the compound. The heterocyclic carboxylic acids have incorporated new functionalities into coordination compounds. The nitrogen atom having proton donor and acceptor ability in the ligand, and the carboxylate oxygen coordinated with the metal ion results in the formation of exact networks having targeted topologies [18-21].

Based on a thorough literature search and to the best of our knowledge, there has been no report of metal complexes synthesized by a combination of 2,3-dihydroxybutanedioc acid with 4-hydroxybenzoic acid, and also 1,4-benzenedicarboxylic acid with 4-hydroxybenzoic acid ligands using Co(II), Cu(II), Fe(II), and Zn(II) ions. Thus, we herein report the preparation of Co(II), Cu(II), Fe(II), and Zn(II) ions coordination polymers using mixed 2,3-dihydroxybutanedioic acid (DHB), 1,4-benzenedicarboxylic acid (BDC), and 4-hydroxybenzoic acid (4-HBA) ligands by a solvent-based method of refluxing, and a solvent-free method of grinding.

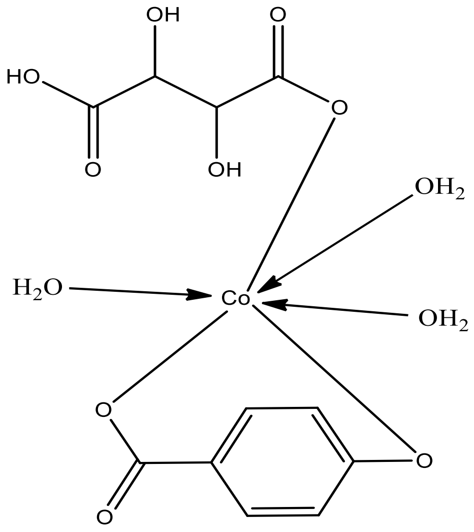
1. Materials And Methods

1.1. Materials used for syntheses

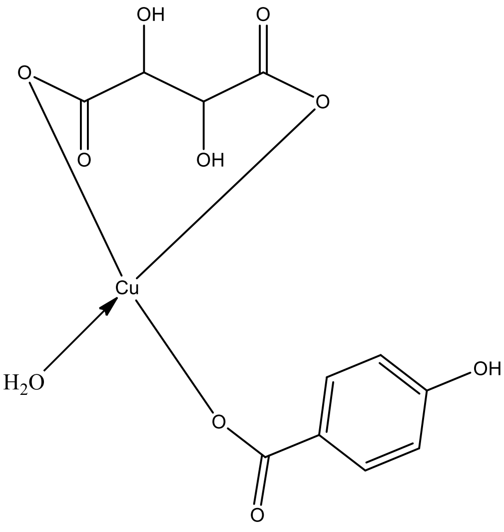
The ligands: 2,3-Dihydroxybutanedioic acid (95 %), 4-hydroxybenzoic acid (95 %), and 1,4-benzenedicarboxylic acid (95 %), were purchased from Sigma Aldrich Co., Germany and used as received. The metal salts: cobalt(II) chloride tetrahydrate ($CoCl_2.6H_2O$, 97 %), copper(II) sulphate pentahydrate ($CuSO_4.5H_2O$, 97 %), zinc(II) sulphate tetrahydrate ($ZnSO_4.6H_2O$, 98 %) and iron(II) chloride tetrahydrate ($FeCl_2.4H_2O$, 98 %) were also purchased from Sigma Aldrich Co., Germany, and used as received.

1.2. Synthesis of the complexes

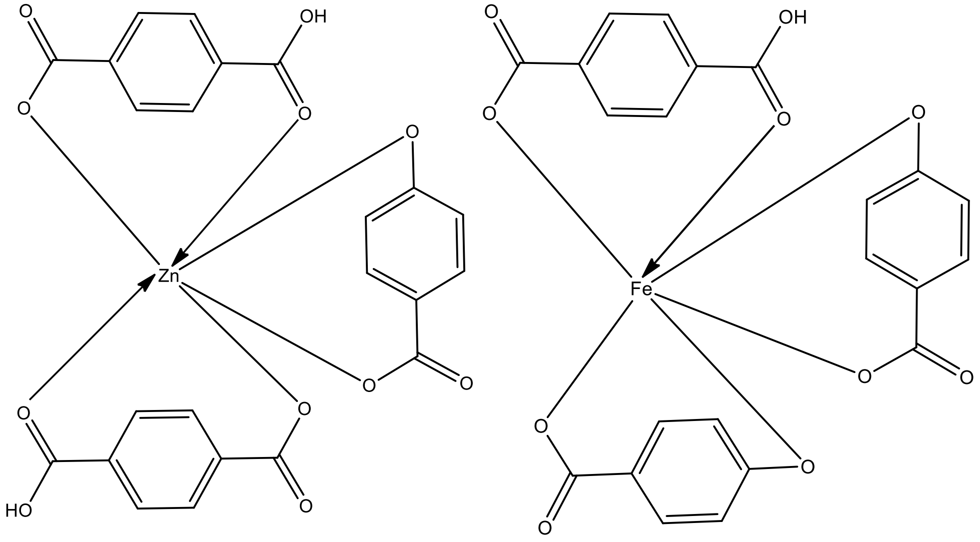
[Co(DHB)(4-HBA)] (**1**) was synthesized by a solvent-based method of refluxing which involved dissolving the ligands,2,3-dihydroxybutanedioic acid (DHB) in dimethylformamide (DMF) and 4-hydroxybenzoic acid (4-HBA) ethanol, and mixed with an aqueous solution of $CoCl_2.6H_2O$ in mole ratios of 1:1:1. [Cu(DHB)(4-HBA)] (**2**) was prepared by a solvent-free method of grinding, in a mortar using a pestle, the ligands and $CuSO_4.5H_2O$ in mole ratios of 1:1:1 in the absence of a solvent system.[Zn(BDC)(4-HBA)] (**3**) and [Fe(BDC)(4-HBA)] (**4**) were prepared by dissolving terephthalic acid (BDC) and 4-hydroxybenzoic acid (4-HBA) in DMF and ethanol respectively and thereafter mixed with an aqueous solution of $ZnSO_4.6H_2O$ and $FeCl_2.4H_2O$. The reaction was carried out with molar ratios of 1:1:1 for the ligands and metal salts, under reflux for 2 hours at 80 ŋC (Fig. 1).



Complex **1**



Complex **2**



Complex **3** Complex **4**

Figure 1. Proposed structures for Complexes **1-4**.

1.3. Characterization of the complexes

Thermal analysis of the complexes formed was carried out using a TGA 4000 Thermogravimetric analyzer PerkinElmer in the temperature range of 30-950 ŋC, while the melting points of the complexes were determined using a Gallen-Kamp melting point apparatus.

Fourier transform Infrared (FTIR) spectra of the complexes were obtained using a Thermo scientific NICOLET iS5 Spectrometer and the UV-visible spectra were obtained from a Cary 100 UV-Vis spectrophotometer. X-ray diffraction (XRD) analysis of the complexes was carried out using an Empyrean Panalytical X-ray Diffractometer, while the elemental analysis of the compounds was done using a Perkin-Elmer 2400 series II C, H, N analyzer.

2. Results And Discussion

2.1. Spectroscopic characterization of the complexes

Fourier transform Infra-red (FTIR) spectra of the prepared complexes **1**, **2**, **3**, and **4** are presented in Fig. 2. The $\nu_{asym}(COO^-)$ and the $\nu_{sym}(COO^-)$ peaks were observed at 1682 and 1397 cm^{-1}, respectively, for complex **2** and at 1609 and 1381 cm^{-1} for complex **1**. Complex **3** exhibited $\nu_{asym}(COO^-)$ and $\nu_{sym}(COO^-)$ peaks at 1653 and 1362 cm^{-1} while in the complex **4** the peaks were observed at 1685 and 1387 cm^{-1}, respectively. The carboxylate group $\Delta\nu=[\nu_{asym}-\nu_{sym}]$ values in the complexes were found to be 228, 285, 291, and 298 cm^{-1} for complexes **1-4**, respectively, suggesting a monodentate mode of coordination [22,23]. The peaks observed at 428, 441, 488, and 450 cm^{-1} in complexes **1-4**, respectively, are attributed to the metal oxygen ν(M-O) bending vibration [23-25].

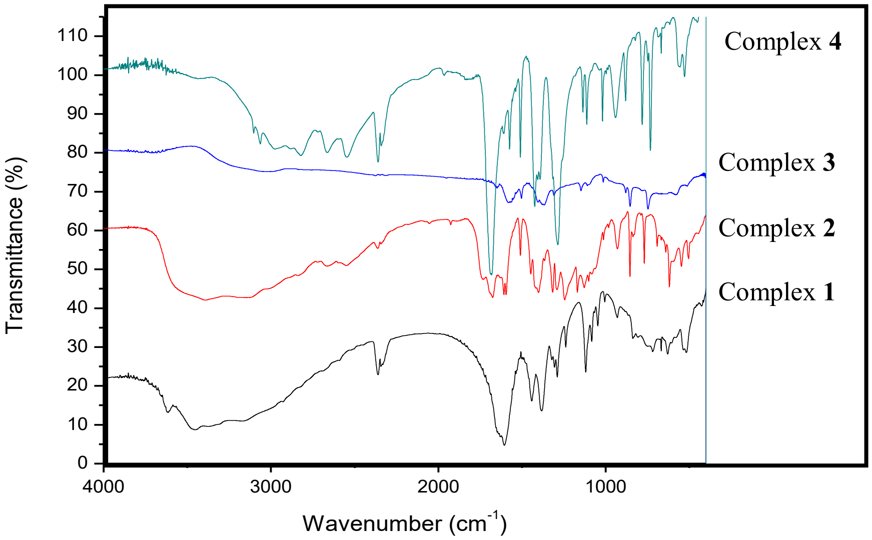


Figure 2. FTIR Spectra of complexes **1-4.**

UV-vis spectrophotometric analysis of the complexes (Table 1) showed a shift from the n-π* transition values observed in the ligands. This may be attributed to the presence of the metal ion in the complexes. The n-π* transition for complex **1** observed at 365 nm (27397 cm^{-1}) showed a shift from the 360 nm (27778 cm^{-1}) observed in the DHB ligand used. The complex **4** showed the n-π* transition at 361 nm (27701 cm^{-1}) while the π-π* transition was observed at 330 nm (30303 cm^{-1}) indicating a shift from the 327 nm (30581 cm^{-1}) observed in the BDC ligand [26,27].

Table 1. UV-visible spectroscopy result of complexes **1-4**

Compound/ ligand	Wavelength (nm)	Energy (cm^{-1})	Assignment
BDC	327	30581	π-π*
DHB	360	27778	n-π*
4-HBA	390	25641	n-π*
Complex **1**	365	27397	n-π*
Complex **2**	328	30488	π-π*
Complex **3**	324	30864	π-π*
Complex **4**	330	30303	π-π*
	361	27701	n-π*

2.2. Thermal properties of the complexes

Thermal decomposition studies of the complexes carried out in a nitrogen atmosphere by thermogravimetric (TG) and differential thermogravimetric (DTG) analysis showed that complex **1** was stable upon initial heating from 30 ŋC until 200 ŋC (Fig. 3a). An initial 9.406 % mass loss attributed to the removal of three coordinated water molecules [28,29] was observed upon continued heating of the complex **1** from 200 ŋC to 517 ŋC. A two-step decomposition process was observed from the TGA curve with the first decomposition process observed between 200 ŋC to 517 ŋC, while the second decomposition process was observed between 517 ŋC to 775 ŋC. Complex **2** also showed a two-step decomposition process with a 1.883 % mass loss observed from 80 ŋC-352.68 ŋC (Fig. 3b) which is attributed to the presence of lattice water molecule in the complex [30,31] while the second decomposition process was observed between 352 ŋC to 590 ŋC with a 91.868 % weight loss.

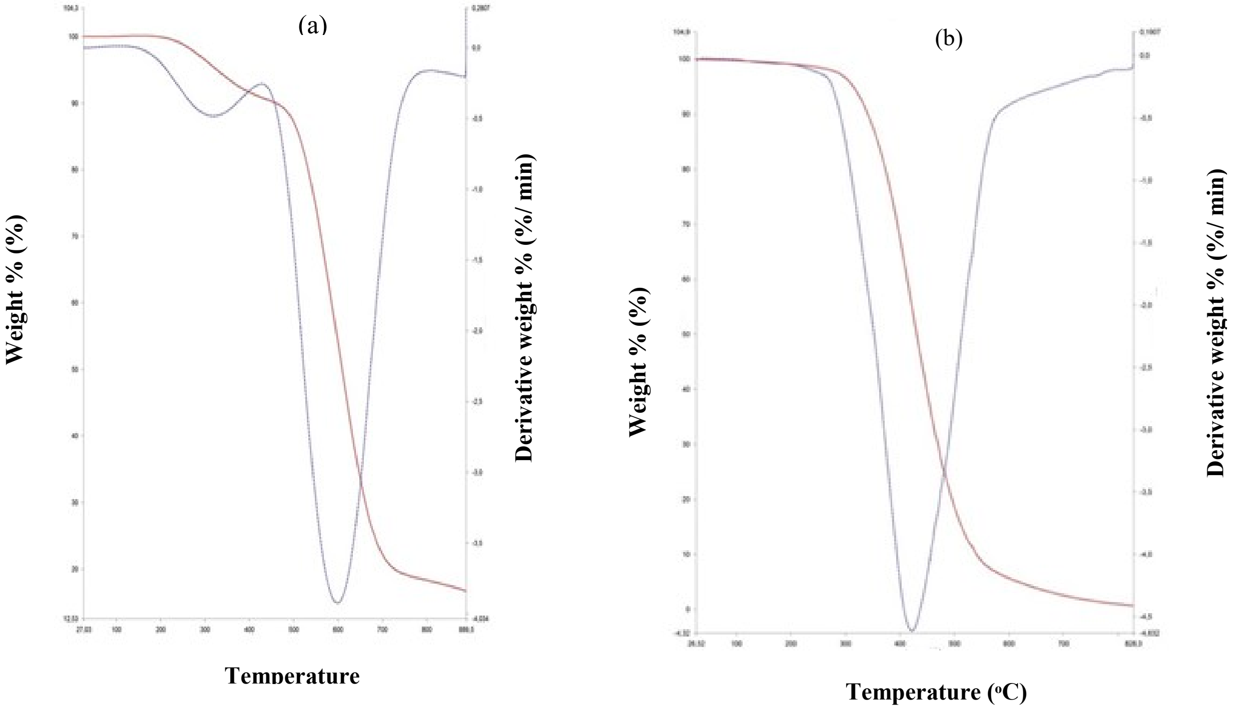


Figure 3. TGA and DTA curves of (a) complex **1** and (b) complex **2**.

The TG curves of complex **3** and complex **4** are given in Fig. 4a and 4b. TG curve of complex **3** showed an initial mass gain of 2.963 %. This may be attributed to absorption by complex **3** in the temperature range of 30 ŋC-130 ŋC. The curve showed that the weight loss begins at a temperature of about 207 ŋC for complex **3** (Fig 3a) and continues until about 405 ŋC with a weight loss of 91.758 %. A 9.863 % mass loss was observed thereafter between a temperature of 410-570 ŋC which signifies the decomposition of the remaining backbone component of the complex [29,30]. Complex **4** showed stability with a rise in temperature (Fig. 4b) till about 208 ŋC when decomposition began and continues till about 450 ŋC with a weight loss of 89.958 % [31,32].

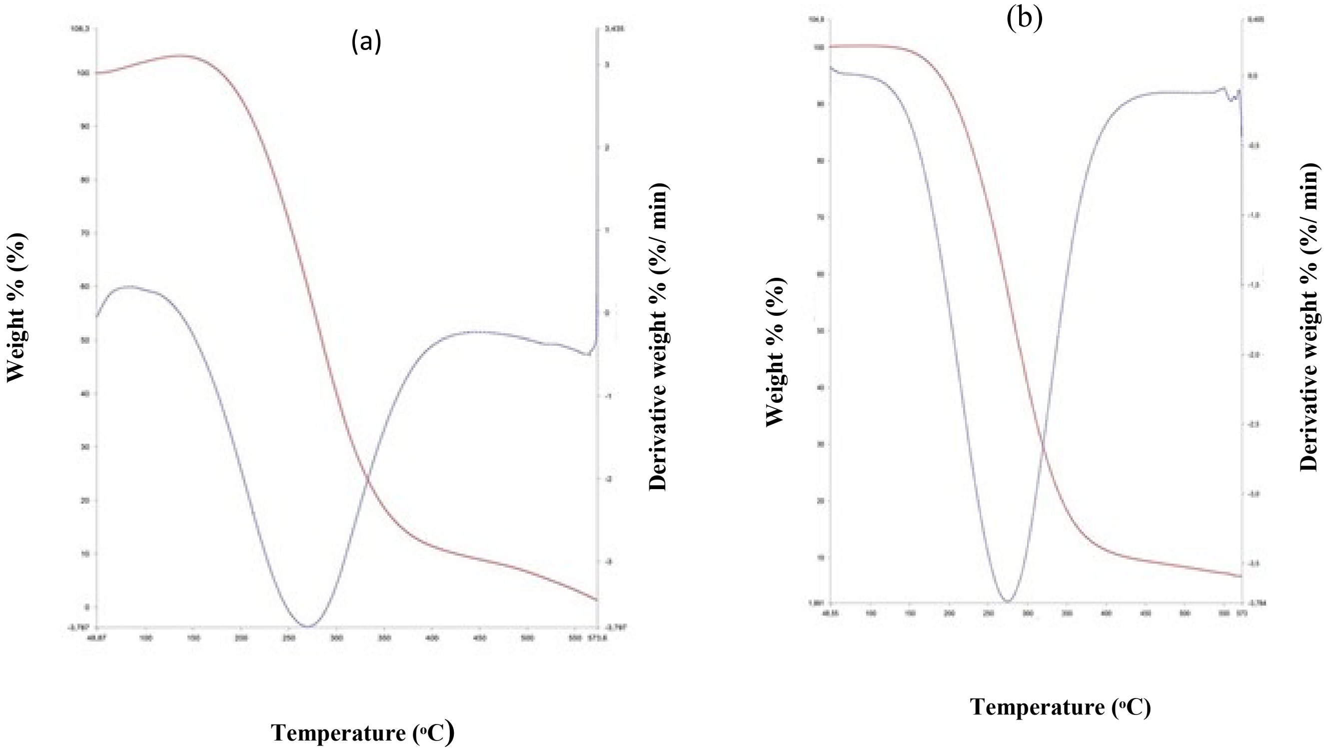


Figure 4. TGA and DTA curves of (a) complex **3** and (b) complex **4**.

The onset decomposition temperature of complexe **1** was observed to differ widely from that of complex **2**, while complexes **3** and **4** were observed to have very close onset decomposition temperature. The DTA curve of complex **1** shows a medium broad T_{DTG} observed between 170-420 ŋC which corresponds to the 9.406 % weight loss observed in the TG curve and accompanied by a sharp T_{DTG} observed at about 600 ŋC corresponding to the second step decomposition process observed in the TG curve (Fig. 3a). Complex **2** showed a very weak T_{DTG} peak observed around 165 ŋC and a sharp peak (T_{DTG}) observed at 420 ŋC which compliments the observed two-step decompositions in the TG curve of complex **2** (Fig. 3b). The T_{DTG} curve of complex **3** also showed a rise at about 70 ŋC indicating the occurrence of absorption in the complex around 70 ŋC as observed in the TG curve (Fig. 4a). A weak T_{DTG} peak observed between 430-570 ŋC also compliments the weight loss observed in the TG curve in the same range.

2.3. Melting points of the complexes

The melting points of the complexes prepared are presented in Table 2. The melting point of complex **1** was observed to be 380 ŋC which is much higher than the parent ligands used, but lower than the decomposition temperature observed in the TG curve. Complex **2** showed a much lower melting point at 217 ŋC compared to the complex **1** which was synthesized using the same parent ligands. Complexes **3** and **4** had melting temperatures of 352 ŋC and 384 ŋC, respectively. These melting temperatures were observed to be close to the major TG decomposition temperatures of complexes **3** and **4**.

Table 2. Melting points of complexes **1-4**.

Compound	**Melting Points (ŋC)**
Complex **1**	380
Complex **2**	217
Complex **3**	352
Complex **4**	384
4-hydroxybenzoic acid (4-HBA)	214
1,4-benzenedicarboxylic acid	300
2,3-dihydroxybutanedioic acid (DHB)	172

2.4. Powder X-ray diffraction analysis of the synthesized complexes

The X-ray diffraction (XRD) analysis of the complexes showed the non-crystalline nature of the compounds. XRD analysis of complex **1** showed peaks at 20.7062°, 1243; this was observed to shift to lower intensity of 20.7850, 898 in complex **2**. Peaks observed at 38.9048°, 650 and at 21.2839°, 615 in complex **3** appeared at higher intensities of 38.7473°, 821 and 20.6799°, 1205, respectively in complex **4**. Fig. 5 presents the XRD patterns of the complexes.

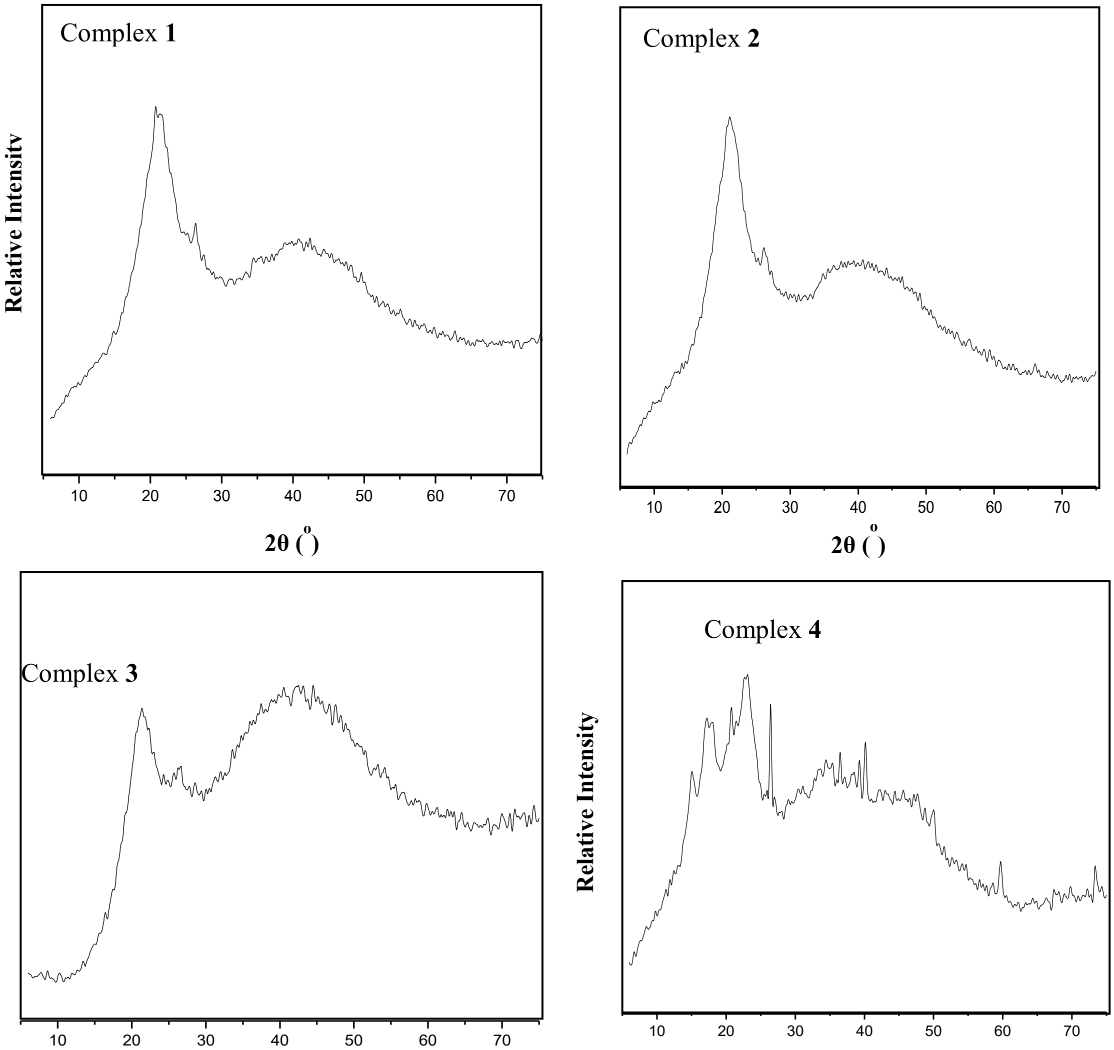


Figure 5. X-ray diffraction of complexes **1-4**.

2.5. Elemental analysis result

The C, H, N elemental analysis of the synthesized complexes showed the percentage carbon (C) and hydrogen (H) present in the compounds. Complex **1** showed a percentage found in C and H to be 32.79 and 3.45, respectively, while the percentage calculated was observed to be 33.18 and 3.80, respectively, which correlates with the presence of three water molecules in the structure as reported under the thermal analysis. Table 3 presents the results of elemental analysis of the complexes.

Table 3. C and H analysis of complexes **1-4**

		C	**H**
Complex **1**	% Found	32.79	3.45
	% Calc.	33.18	3.80
Complex **2**	% Found	35.86	2.74
	% Calc.	36.02	3.02
Complex **3**	% Found	50.84	2.32
	% Calc.	51.95	2.65
Complex **4**	% Found	52.86	2.47
	% Calc.	53.48	2.66

3. CONCLUSION

The synthesis of four mixed carboxylate ligand-metal complexes, [Co(DHB)(4-HBA)] (**1**); [Cu(DHB)(4-HBA)] (**2**); [Zn(BDC)(4-HBA)] (**3**); and [Fe(BDC)(4-HBA)] (**4**), their thermal and spectroscopic properties are herein described. Thermal studies of complexes **1** and **2** showed a two-step decomposition process. Complex **4** was observed to be stable with an increase in temperature till 208 ŋC. These complexes can, thus, find applications in relatively high thermal processes in which decomposition of the complexes is not desired.

Acknowledgement The authors are grateful to Dr. A.C. Tella, of the Laboratory of Inorganic and Material Chemistry, Dept. of Chemistry, University of Ilorin, for the contributions towards this research.

Özet

Co (II), Cu (II), Fe (II) ve Zn (II) iyonlarına dayalı koordinasyon bileşiklerinin çözücü içeren ve çözücü içermeyen yöntemler ile ligand olarak tereftalik asit (BDC), tartarik asit (DHB) ve 4-hidroksibenzoik asit (4-HBA) kullanılarak sentezi rapor edilmiştir. Komplekslerin 30 ŋC-950 °C sıcaklık aralığında gerçekleştirilen termal çalışmaları, [Co(DHB)(4-HBA)] kompleksi için 200 ŋC-517 ŋC arasında gözlenen % 9.406 kütle kaybını ve [Cu(DHB)(4-HBA)] kompleksi için 80 ŋC-352.68 ŋC arasında gözlenen % 1.883 kütle kaybını göstermiştir.

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