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SYNTHESES AND STRUCTURAL ANALYSES OF HETERONUCLEAR HEXACYANOMETALLATE(III) COMPLEXES WITH 4-(2-AMINOETHYL) PYRIDINE

Seray KEKEÇ[1,*], Güneş Süheyla KÜRKÇÜOĞLU[1]

[1] Department of Physics, Faculty of Science, Eskişehir Osmangazi University, Eskişehir, Türkiye

ABSTRACT

In this study, the complexes, given with the formula $(NH_4)[Cd(\mu\text{-}4aepy)_2Fe(\mu\text{-}CN)_2(CN)_4]_n$ and $(NH_4)[Cd(\mu\text{-}4aepy)_2Co(\mu\text{-}CN)_2(CN)_4]_n$,, obtained by using cadmium(II), 4-(2-aminoethyl)pyridine (4aepy) and hexacyanoferrate(III) or hexacyanocobaltate(III) were synthesized as powder. Structural characterizations of the obtained compounds were investigated by vibration (FT-IR and Raman) spectroscopy, elemental, thermal and powder-XRD techniques. Vibration spectra of the complexes were recorded between 4000-225 cm^{-1} and were confirmed by the results of thermal and elemental analyses. The structural properties of the complexes were investigated by considering the characteristic bands of the ligands from the vibration spectra. Additionally, vibration spectra of the complexes were observed two different ν(CN) absorption bands which act as bridge and terminal. The spectral data show that the characteristic bands of 4aepy were shifted to lower and high frequencies. These shifts arise due to bounding metal and coupling of metal-ligand vibration modes. Thermal behaviors of the complexes are investigated between the temperature ranges of 30-1000 °C in static air atmosphere.

Keywords: Hexacyanoferrate(III); Hexacyanocobaltate(III); 4-(2-aminoethyl)pyridine; Coordination polymer.

1. INTRODUCTION

In recent years, attention has been focused on the design and synthesis of new coordination polymers due to the infinite variety of their structural types, architectures, and essential properties and applications [1-5]. In previous years by synthesizing coordination compounds, Werner, Jorgensen, and other scientists investigated their structures, and various theories were devised to explain them. Determining the structure and properties of metal complexes has been a subject of research for a very long time [6-10].The creation of new coordination polymers, formed from organic binders and metal ions, is of great interest due to their impressive structure and many potential uses (adsorption, sensor, catalysis, luminescence, magnetism, sorption, gas separation, conductivity, etc.) [11-19]. Although significant advancement has been made in the synthesis of coordination polymers, their controlled synthesis with the desired structures and properties is still a formidable challenge. The characteristics of ligands, such as flexibility and length, can influence the structural diversity of complexes and the dimensionality of compounds. Additionally, the reaction conditions, solvent, temperature, pH, etc. can affect the ultimate structure of coordination polymers. In recent years, synthesis techniques and designs of heterometallic coordination polymers have become the focus of numerous studies.

Cyanide complexes were among the initially discovered coordination compounds to be prepared and explored [20-24], in coordination and organometallic chemistry, they continue to be extensively investigated. Organometallic and coordination chemistry makes use of the skill of cyanide complexes to bond to various central atoms [24-26] and supramolecular chemistry to construct 1-dimensional, 2-dimensional, and 3-dimensional structures [27-32]. Metal cyanide complexes are typically not considered organometallic, despite the fact that cyanide as a monodentate ligand always appears to have a carbon atom as the donor atom.

A previous study reported the synthesis, vibration (FT-IR and Raman) spectra, thermal, elemental, powder and single-crystal X-ray diffraction studies of hexacyanoferrate(III) or hexacyanocobaltate(III) of 4-(2-aminoethyl)pyridine (4aepy) [33]. In this study, Fe(III)/Co(III) cyanide complexes and 4-(2-aminoethyl)pyridine ligand were used for the preparation of these complexes formulated as $(NH_4)[Cd(\mu\text{-}4aepy)_2Fe(\mu\text{-}CN)_2(CN)_4]_n$ and $(NH_4)[Cd(\mu\text{-}4aepy)_2Co(\mu\text{-}CN)_2(CN)_4]_n$ (where 4aepy = 4-(2-aminoethyl)pyridine, abbreviated hereafter as Cd-M'-4aepy) in powder form. The structures of the obtained complexes as powder were elucidated by vibration (FT-IR and Raman) spectra, elemental, thermal analyses and powder XRD patterns. As a result, it was seen that the complexes obtained as single crystals in the literature were similar to their structures [33].

2. EXPERIMENTAL

2.1. Materials and Instrumentation

2.1.1. Chemicals

Potassium hexacyanometallate(III) ($K_3[Fe(CN)_6]$, Merck, 99% and $K_3[Co(CN)_6]$, Acros, 95%), cadmium(II)chloride hemipentahydrate ($CdCl_2{\cdot}2.5H_2O$, Acros, 99%) and 4-(2-aminoethyl)pyridine ($C_7H_{10}N_2$, TCI, 95%) were purchased from commercial sources and were used as received.

2.1.2. Characterization of the complexes

Fourier Transform Infrared Spectrometer (FT-IR/Perkin Elmer Spectrum 100) was taken with Attenuated Total Reflection (ATR) at a resolution of 4 cm^{-1} in between 4000-225 cm^{-1}. Elemental analyses (C, H and N) were taken on the LECO CHNS-932 analyzer. Raman spectra were recorded in the range of 4000-225 cm^{-1} with a Raman spectrum device (Bruker Senterra Dispersive Raman) using laser excitation at 785 nm. X-ray diffractometer (Panalytical Emperian) with Cu-Kα radiation was used for powder X-ray diffraction (PXRD) patterns. Thermal analyzes of the complexes (PerkinElmer Diamond TG/DTA Thermal Analyzer) were recorded in a static air atmosphere with a heating rate of 10 °C min^{-1}.

2.2. Syntheses

2.2.1. Syntheses of complexes Cd-M'-4aepy

The synthesis of complexes Cd-Fe-4aepy and Cd-Co-4aepy were prepared by the method mentioned in this literature [33]. Solutions were filtered and stored at room temperature. Complexes Cd-Fe-4aepy and Cd-Co-4aepy obtained in powder form after a few days. Yellow powders of Cd-Fe-4aepy were obtained in 65% yield. Anal. Calcd. for $C_{20}H_{24}CdFeN_{11}$ (Cd-Fe-4aepy) (M_w= 586,73 g mol^{-1}): C 40.36, H 4.41, N 26.35%; found C 40.94, H 4.22, N 26.26% and yellow powders of Cd-Co-4aepy were obtained in 69% yield. Anal. Calcd. for $C_{20}H_{24}CdCoN_{11}$ (Cd-Co-4aepy) (M_w= 589,82 g mol^{-1}): C 39.24, H 4.26, N 25.57%; found C 40.03, H 4.10, N 26.02%.

3. RESULTS and DISCUSSION

3.1. Vibration spectra of complexes Cd-Fe-4aepy and Cd-Co-4aepy

Figures 1 and **2** represent the vibration (FT-IR and Raman) spectra. **Table 1** lists the wavenumbers of 4aepy ligand observed in the FT-IR spectra of the complexes [33-35]. As can be seen in **Figures 1** and **2**, the presence of 4aepy bands in the FT-IR and Raman spectra of the complexes indicates the presence of 4aepy and cyanide in the complexes. The 4aepy ligand was used to evaluate the spectral analyses of each complex individually.

There is free ammonium molecule in the structures of the complexes. In complexes Cd-Fe-4aepy and Cd-Co-4aepy, stretching ν(NH) vibrations of free ammonium molecule were observed, with asymmetric ν(NH) stretching at 3347 cm^{-1} and symmetric ν(NH) stretching at 3274 cm^{-1} for Cd-Fe-4aepy, and asymmetric ν(NH) stretching at 3329 cm^{-1} and symmetric ν(NH) stretching at 3288 cm^{-1} for Cd-Co-4aepy [33]. In FT-IR spectra of Cd-Fe-4aepy and Cd-Co-4aepy was observed that the symmetrical and asymmetric ν(NH) stretching vibrations of the ammonium cation and the free ammonium molecule had shifted to a lower frequency. The four vibration modes of ammonium ions are separated into two modes of stretching and two modes of bending. Only two of these four modes (asymmetric stretching and bending) are IR active. Each of the four modes is Raman active. T_d is the symmetry group of an isolated NH_4^+ cation. The absorption bands of the NH_4^+ group are between 3300 - 3030 cm^{-1}, which comply to the stretching vibration [33, 36]. The broad band at 3232 cm^{-1} in the FT-IR and the weaker band at 3162 cm^{-1} in the Raman spectrometer indicate NH_4 ion's asymmetric and symmetric stretching vibrations. The band ascertained in the Raman spectrum at 1688 cm^{-1} comply to the NH_4 ion's asymmetries attributes symmetrical bending of NH_4 to bands at 1391 cm^{-1} in FT-IR and 1380 cm^{-1} in Raman [33, 37].

3.1.1. 4-(2-aminoethyl)pyridine (4aepy) vibrations

Since 4aepy contains two nitrogen atoms that donate electrons, it is thought that bonding will occur between the two nitrogen atoms of 4aepy and the transition metals. When 4aepy is coordinated with transition metal ions, large changes are observed in the vibration 4aepy bands [33, 38].

The 4aepy absorption bands at 3359 cm^{-1} and 3287 cm^{-1} are due to the asymmetric and symmetric stretching ν(NH_2) vibrations. Absorption bands of stretching ν(NH_2) vibrations are observed in the region 3347-3274 cm^{-1} and shifted lower in frequency than 4aepy in both complexes. The amino group of 4aepy shifts the positions of the absorption ν(NH) bands to lower wavenumbers via the nitrogen atom. The deformation δ(NH_2) band of 4aepy was defined at 1601 cm^{-1} and shifts to higher frequencies were observed in the complexes [33].

3.1.2. Cyanide vibrations

Stretching ν(CN) vibrations, which are particularly essential for complexes, have been found to be carried to the high or low frequency region in complexes. The positions of the stretching ν(CN) bands in the FT-IR spectrum of complexes containing cyanide ligands are an important tool to determine the number of cyanide groups present in the complexes and the type of terminal or bridging. The stretching ν(CN) vibrations of a terminal cyanide ligand range from 2000 - 2100 cm^{-1}, while the 2100 - 2200 cm^{-1} range is characteristic of a cyanide ligand acting as a bridge and a terminal cyanide ligand participating in hydrogen bond interactions [33, 39-42]. Cd-Fe-4aepy and Cd-Co-4aepy include cyanide stretching band is ascertained in the FT-IR spectra at 2130 cm^{-1} for Cd-Fe-4aepy, 2124 cm^{-1} for Cd-Co-4aepy [33].

Table 1. The FT-IR and Raman wavenumbers of the 4aepy in complexes (cm^{-1}).

Assignments [33-35]	**4aepy**	**Cd-Fe-4aepy**		**Cd-Co-4aepy**	
		FT-IR	**Raman**	**FT-IR**	**Raman**
$\nu_{as}(NH_2)$	3359 m	3347 vw	3330 vw	3329 w	3335 vw
$\nu_s(NH_2)$	3287 m	3274 vw	3278 w	3285 vw	3285 vw
$\nu(NH_2)$	3181 vw	3187 vw	3183 vw	3185 vw	3199 w
ν(CH)	3072 w	-	3062 w	-	3075 vw
ν(CH)	3025 w	3019 sh	3000 w	3005 vw	3034 vw
ν(CH)	2993 vw	2998 m	2965 m	2992 vw	-
$\nu(C\text{-}CH_2\text{-}NH_2)$	2931 m	2973 m	2930 m	2976 w	2972 vw
$\nu(C\text{-}CH_2\text{-}NH_2)$	2856 w	2878 m	2862 w	2879 m	2864 vw
ν(C=N)	1663 sh	1668 vw	-	1667 vw	1645 w
$\delta(NH_2)$	1601 vs	1615 s	1615 w	1616 s	-
ν(C=C)+ν(C=N)	1556 s	1561 vw	1553 vw	1558 vw	1575 w
$\delta(CH_2)$	1496 w	1480 sh	1505 vw	1500 sh	1500 w
$\delta(CH_2)$	1462 vw	1467 m	1462 vw	1471 m	1469 vw
ν(C=N)+ν(C=N)	1439 w	1440 m	1445 w	1426 m	1432 w
w(CH)	1414 vs	1425 s	-	1424 s	1429 w
t(CH)	1385 w	1390 sh	-	-	1384 w
ν_{ring}+ δ(CH)	1360 w	1367 vw	1357 w	1367 w	1367 w
$t(NH_2)$	1321 w	1328 vw	1326 w	1322 vw	1325 w
ν_{ring}	1218 s	1216 m	1213 m	1221 m	1211 w
ν_{ring}+ δ(CH)	1130 w	1101 vw	1131 vw	1131 vw	1146 vw
δ(CH)	1069 m	1068 m	1069 w	1069 m	1083 w
Ring breathing	1016 w	1034 vs	1013 m	1035 s	1034 vw
Ring breathing	992 m	994 sh	-	1013 m	1013 w
Ring deformation	962 w	971 s	926 w	969 m	947 vs
$t(CH_2)$	837 w	835 sh	876 w	838 sh	840 m
Ring breathing	800 m	811 m	817vw	815 m	815 m
γ(CH)	766 w	779 m	782 m	785 m	786 m
$r(NH_2)$	594 w	608 w	592 w	604 sh	607 w
$r(NH_2)$	581 w	586 m	-	590 m	592 vw
γ_{ring}	514 m	516 s	513 w	521 vw	520 m
γ(CC)	493 w	508 s	468vw	509 m	495 w

Abbreviations used: ν, stretching; δ, deformation; w, wagging; t, twisting; r, rocking; s, strong; m, medium; w, weak; sh, shoulder; v, very.

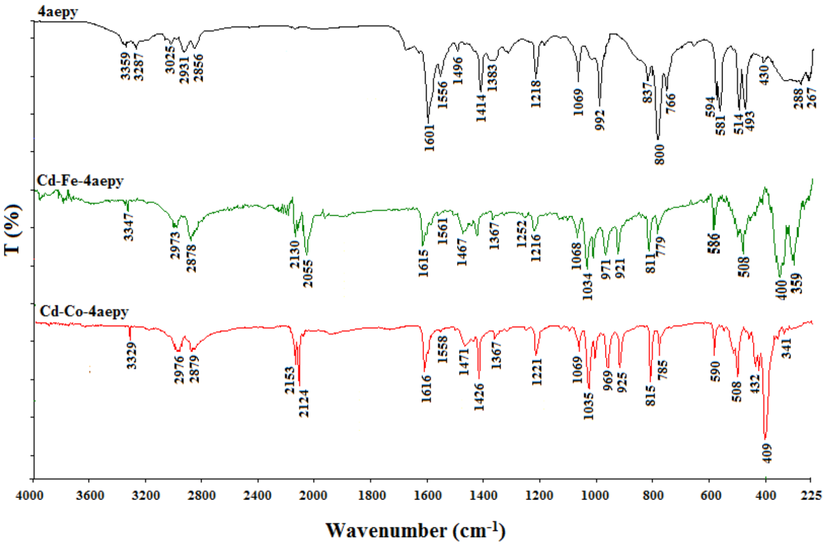


Figure 1. The FT-IR spectra of 4aepy and complexes.

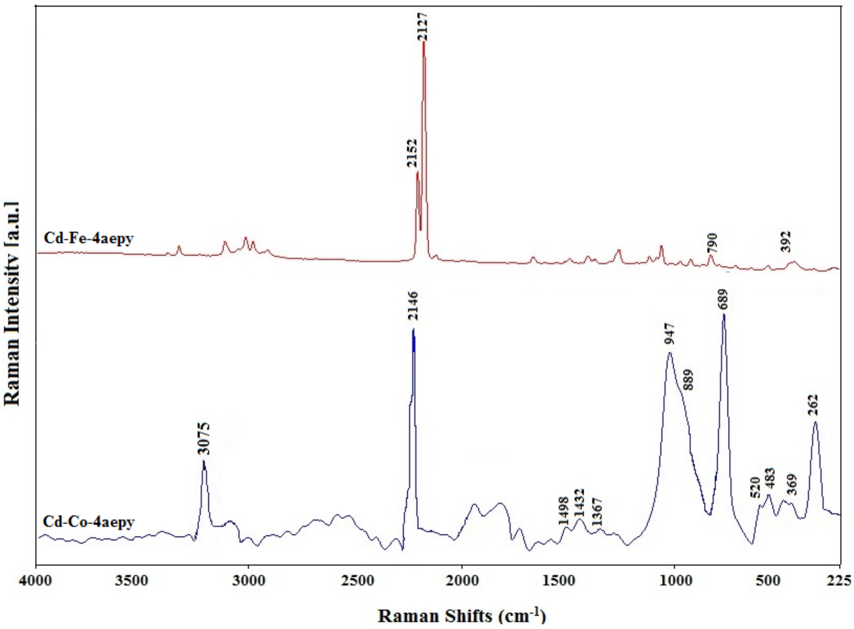


Figure 2. Raman spectra of complexes.

3.2. PXRD diffraction analyses

PXRD diffraction model of **1** and **2** are given in **Figure 3**.

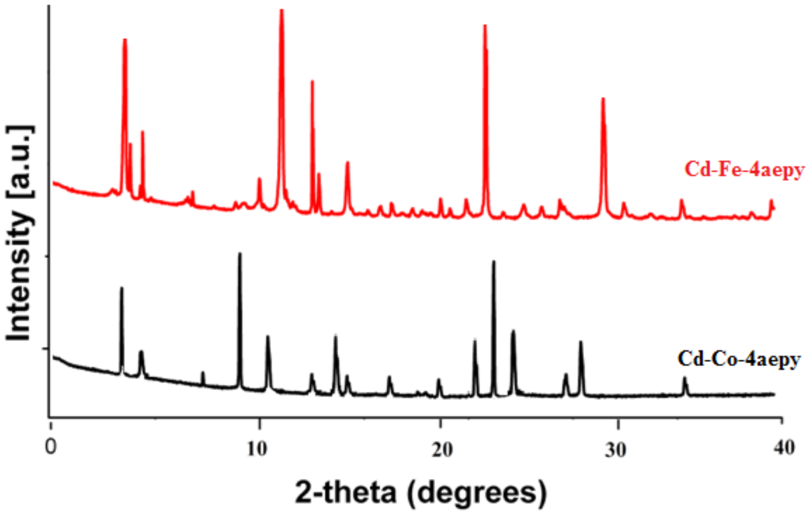
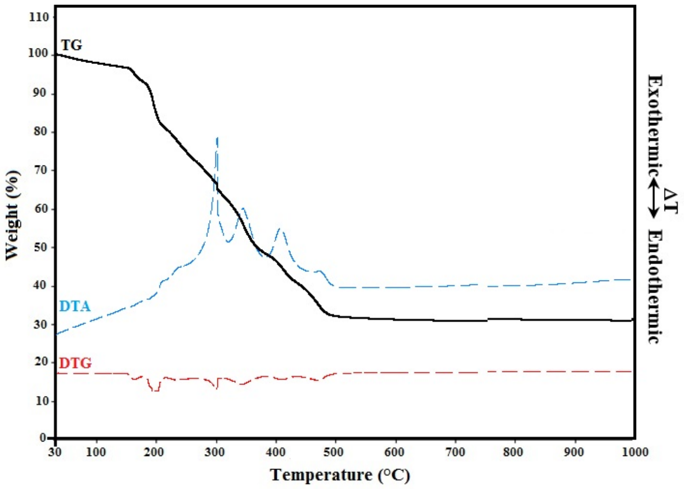


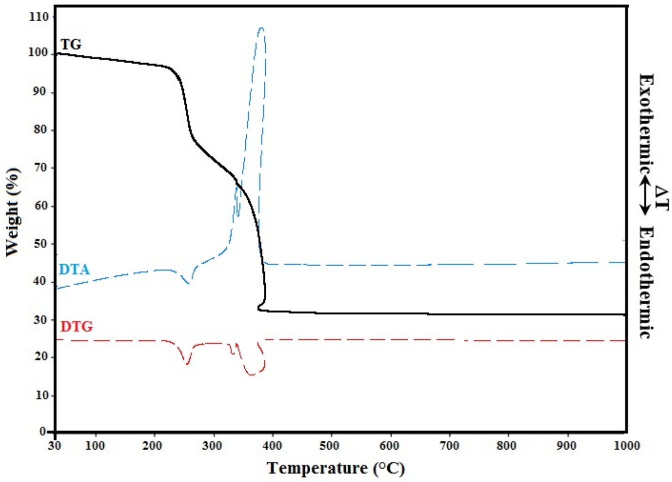
Figure 3. The PXRD models of complexes.

3.3. Thermal analyses

The thermal analyses of the complexes were carried out in static air environment with a heating rate of 10 °C min^{-1}, and the temperature was increased from 30 - 1000 °C at a rate of 10 °C min^{-1}. The TG, DTA and DTG curves are shown in **Figure 4**.



(a)



(b)

Figure 4. The TG, DTG and DTA curves of (a) Cd-Fe-4aepy, (b) Cd-Co-4aepy.

In the first stage of Cd-Fe-4aepy and Cd-Co-4aepy**,** the complexes lose one NH_4 molecule in the temperature range of 41-155 °C [found (calcd.): 3.1% (3.05%)] for Cd-Fe-4aepy and 44-208 °C [found (calcd.): 2.93% (3.05%)] for Cd-Co-4aepy. The second stage is related to release one 4aepy ligand as a strong endothermic peak on the DTA curve between 155 and 267 ºC with a mass loss of 23.01% (calcd. 21.70%, $DTA_{max.}$ = 217 ºC) for Cd-Fe-4aepy and between 208 and 289 ºC with a mass loss of 22.77% (calcd. 20.71%, $DTA_{max.}$ = 268 ºC) for Cd-Co-4aepy. In the last stage between 267 and 578 °C temperature range for Cd-Fe-4aepy and between 289 and 583 °C temperature range for Cd-Co-4aepy, strong exothermic peak ($DTA_{max.}$= 317 ºC for Cd-Fe-4aepy and 363 ºC for Cd-Co-4aepy) is associated with the removal of one 4aepy and six cyanide ligand from the structure and leading finally to the $CdO+Fe_2O_3$ for Cd-Fe-4aepy and $CdO+Co_3O_4$ for Cd-Co-4aepy. The endothermic peak at 905 ºC is related to the conversion of Co_3O_4 to CoO in Cd-Co-4aepy. The final solid products of thermal decompositions, $CdO+Fe_2O_3$ for Cd-Fe-4aepy and CdO+CoO for Cd-Co-4aepy, were identified by FT-IR spectroscopy (found: 43.37, calcd.: 47.17% for Cd-Fe-4aepy and found: 45.38, calcd.: 47.17% for Cd-Co-4aepy). When the data obtained were examined, it was observed that they were similar to the thermal analyzes of the previously obtained compounds [33].

4. Conclusions

In this study, two hexacyanometallate(III) complexes were synthesized with different bimetallic (Cd(II), Fe(III) or Co(III)) and 4aepy neutral ligand. It has been observed that the structures of these compounds are similar to the vibration (FT-IR and Raman) spectra, PXRD patterns and thermal analyses results of the complexes examined by the single crystal X-ray method in the literature [33]. The formulas of the complexes from elemental analyses data $(NH_4)[Cd(\mu\text{-}4aepy)_2Fe(\mu\text{-}CN)_2(CN)_4]_n$ and $(NH_4)[Cd(\mu\text{-}4aepy)_2Co(\mu\text{-}CN)_2(CN)_4]_n$. Characteristic bands of 4aepy and cyanide ligands, shifts and splits in their positions were observed in the vibration spectra of the complexes. From the thermal analyses data, it was determined that NH_4 group was separated first from the complexes, then the neutral ligands, then the anionic cyanide ligands decomposed and metal oxides remained at the end of the analyses.

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