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Novel Coordination Compounds Based on 2-Methylimidazole and 2,2′-Dimethylglutarate: Synthesis and Characterization

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Abstract: Two novel complexes, $[Cu_2(\mu\text{-dmg})_2(2\text{-meim})_4]\cdot 5H_2O$ **(1)** and $\{[Cd(\mu\text{-dmg})(2\text{-meim})_2]\cdot H_2O\}_n$ (**2**), with 2-methylimidazole (2-meim) and 2,2′-dimethylglutarate (dmg) as ligands, have been systematically prepared by a conventional method. As a result, organic-inorganic crystalline solids were obtained. Their solid-state structures have been solved with elemental analysis, fourier transform infrared spectroscopy (FTIR) Powder X-ray diffraction (PXRD) and single-crystal X-ray diffraction (XRD). The Cu(II) ions displayed a distorted square planar geometry (CuO_2N_2), while Cd(II) ions showed a distorted octahedral geometry (CdO_4N_2). Complex **1** formeda dimeric structure, in which the Cu(II) ions were bridged by dmg. These dimeric units were extended to the 3D supramolecular structure with hydrogen bonding and π-bonding interactions. Complex **2** formed a 1D polymeric structure, where the Cd(II) ions were linked by dmg ligand to form 1D zig-zag polymer layers which were further extended in 3D supramolecular structure through molecular interactions as complex **1**. Moreover, the thermal properties of the complexes were reported.

Keywords: 2-methylimidazole ligand; Cu(II) complex, Cd(II) complex, Coordination polymers, Supramolecular.

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INTRODUCTION

Supramolecular systems and coordination polymers are quite remarkable in coordination chemistry due to their new topologies and different potentials applications like a luminescence, adsorption, catalysis, gas storage, and magnetism, *etc*. (1–4). In many cases, it is very difficult to establish an interaction between the synthesis conditions and the structural properties (5–7). In determining the chemical properties of the coordination polymer, selected metal ions and organic ligands play the key role (8,9). Molecular interactions such as strong hydrogen bonds and weaker non-covalent interactions such as, $\pi\cdots\pi$, C-H$\cdots\pi$, C-H, van der Waals force in the stability of inorganic complexes have shown to be very significant (10,11). Organic molecules containing carboxylic acid have been used as building blocks in crystal solids (12). When heterocyclic rings containing N-donor atoms such as imidazole, triazole, *etc*., are used as ligands, they determine the size and structure of the inorganic network (13–15). Complexes of 2-methylimidazole (2-meim) with transition metal ions have attracted great importance because of their biological and pharmaceutical activities (13,16). In the past, our group studied various N-donor molecules which are a member of the potentially interesting imidazole ligands (11,17). In this work, new dimeric Cu(II) and polymeric Cd(II) complexes,

$[Cu_2(\mu\text{-dmg})_2(2\text{-meim})_4]\cdot 5H_2O$ (**1**) and $\{[Cd(\mu\text{-dmg})(2\text{-meim})_2]\cdot H_2O\}_n$ **(2)** were synthesized with dmg and 2-meim ligands. These complexes were characterized with FTIR, XRD, elemental analysis, PXRD and thermal analysis techniques (TG/DTA) (Scheme 1).

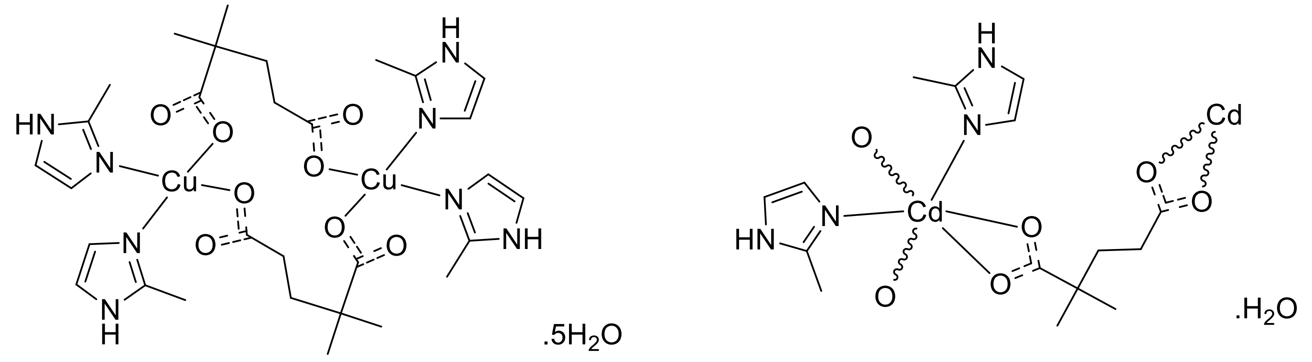
EXPERIMENTAL SECTION

Materials and methods

All chemicals were received and used without any further purification. Elemental analyses (C, H, and N) were performed on a Perkin Elmer 2400C Elemental Analyzer. Powder X-ray diffraction (PXRD) patterns were collected by a Panalytical Empyrian X-ray diffractometer with Cu-Kα radiation (λ= 1.5406 nm). FTIR spectra of the complexes were carried out at room temperature by a Perkin-Elmer FTIR 100 spectrometer in the region of 4000–400 cm^{-1}. The resolution was set up to 4 cm^{-1}, signal/noise ratio was established by 16 scans with Attenuated Total Reflection (ATR). A Perkin Elmer Diamond TG/DTA thermal analyzer was used to record simultaneous TG, DTG and DTA curves in a static air atmosphere at a heating rate of 10 °C min^{-1} in the temperature range 30–1000 °C using platinum crucibles.

Crystallographic analyses

Suitable crystals of **1** and **2** were selected for data collection which was performed on a Bruker D8 QUEST diffractometer equipped with a graphite-monochromatic Mo-K_α radiation at 296 K. The structures were solved by SHELXT and refined by full-matrix least-squares on all F^2 data using SHELXL in conjunction with the OLEX2 graphical user interface (18,19). For all complexes, the anisotropic thermal parameters were refined for non-hydrogen atoms and hydrogen atoms were calculated and refined with a riding model. Molecular drawings were obtained by Mercury and OLEX2 programs (20).The details of data collection and crystal structure determinations are given in Table 1. The crystallographic information file was deposited with the Cambridge Crystallographic Data Centre (CCDC) with the reference number 1826900 for **1** and 1826901 for **2**.



1 **2**

Scheme 1: Molecular structures and coordination modes of dmg and 2-meim ligands in complexes 1 and 2 .

Synthesis of complexes 1-2

A solution of 2,2′-dimethylglutaric acid (0.80 g, 5 mmol) in water (50 mL) was added dropwise with stirring at 80 °C to a solution of $Cu(CH_3COO)_2\cdot H_2O$ (0.99 g, 5 mmol) (**1**) or $Cd(CH_3COO)_2\cdot 2H_2O$ (1.33 g, 5 mmol) (**2**) in distilled water (25 mL). The solutions immediately became suspensions and were stirred for 10 h at 60 °C. Then 2-methylimidazole (2-meim) (1.65 g, 20 mmol) in water (10 mL) was added dropwise to these suspensions. The clear solutions that formed were stirred for 2 h at 60 °C and then cooled to room temperature. The blue (**1**) and colorless (**2**) crystals that formed were filtered and washed with 10 mL of water and dried in air. Anal. Calc. for complex **1** $C_{30}H_{54}Cu_2N_8O_{13}$: C, 41.81; H, 6.32; N, 13.00. Found:C, 41.01; H, 6.26; N, 13.53%. Anal. Calc. for complex **2** $C_{15}H_{24}N_4O_5Cd$: C, 39.79;H, 5.34; N, 12.37. Found: C, 39.94; H, 5.88; N, 12.96%.

RESULTS AND DISCUSSION

Synthesis and characterization

IR spectroscopy was used to elucidate the structures of the synthesized coordination polymers and to identify functional groups. IR spectra showed frequencies in the range 2934-2852 cm^{-1} which are due to ν(C-H) stretching vibrations. ν(N-H) stretching vibrations appear in the range 3152-3263 cm^{-1} for **1** and 3157-3249 cm^{-1} for **2**. The new complexes display a very strong and slightly broad band about at 1500 cm^{-1} due to the combination of C-O and C-C stretches (15). The broad absorption bands of ν(OH) vibrations of water in complexes are detected 3411 and 3433 cm^{-1}, respectively for **1** and **2**. The strong and broad bands corresponding to the asymmetric ($υ_{as}COO^-$) and symmetric ($υ_sCOO^-$) stretching vibrations of carboxylate groups of free dmg ligand approximately at 1694 and 1414 cm^{-1}(21). In the resulting new complexes, these bands are seen at about 1526-1572 cm^{-1} and 1414–1401 cm^{-1} regions respectively.

Description of the crystal structure

Table 1 indicates the crystal data and structural refinement parameters.

Table 1. Crystal data and structural refinement parameters for complexes **1** and **2.**

	1	**2**
Empirical formula	$C_{30}H_{54}Cu_2N_8O_1$	$C_{15}H_{24}CdN_4O_5$
Formula weight (g/mol)	861.89	452.78
Crystal system	Orthorhombic	Monoclinic
Space group	Pbca	P21/n
a (Å)	14.3309 (11)	14.020 (5)
b (Å)	14.1050 (14)	10.644 (4)
c (Å)	40.693 (4)	14.659 (6)
α(°)	90	90
β (°)	90	117.492 (11)
γ(°)	90	90
V (Å3)	8225.6 (13)	1940.6 (13)
Z	8	4
Dc (g cm-3)	1.392	1.550
μ (mm-1)	1.10	1.16
Measured refls.	89930	20572
Independent refls.	8351	3880
Rint	0.086	0.085
R [I>2σ(I)]	0.077	0.070
wR [I>2σ(I)]	0.166	0.181
S	1.27	1.20

$[Cu_2(\mu\text{-dmg})_2(2\text{-meim})_4]\cdot 5H_2O$ (1). The molecular structure of complex **1** consists of dimeric $[Cu_2(\mu\text{-dmg})_2(2\text{-meim})_4]$ units (Figure 1). The complex has the orthorhombic crystal system with the *Pbca* space group (Table 1). According to single crystal X-ray structural analysis, the asymmetric unit of the complex **1** contains two copper ions, two bridging dmg ligands, four 2-meim moieties and five uncoordinated water molecules. Each copper metal ion coordinated to two different dmg ligands by means of the oxygen atoms and two different 2-meim ligands by means of the nitrogen atoms. The geometry of each Cu(II) ion is a distorted square planar geometry (CuO_2N_2). The dmg ligands forms a bridge between the Cu(II) ions by means of the four carboxylate atoms of each dmg to form a 16 membered dimeric $[Cu_2(\mu\text{-dmg})_2]$ unit. These dimeric units are extended to 3D supramolecular structure with intra- and intermolecular hydrogen bonds, C–H···π, C–O···π and π···π interactions (Figure 2).

Table 2: Selected bond distances (Å), angles (°) and hydrogen-bond geometry data for **1.**

Bond Lengths (Å)			
Cu1—O1	2.005 (4)	Cu2—O3	1.967 (4)
Cu1—O5	1.968 (4)	Cu2—O7	1.966 (4)
Cu1—N3	1.992 (4)	Cu2—N7	1.982 (5)
Cu1—N1	1.981 (4)	Cu2—N5	1.983 (5)
Angles (°)			
O5—Cu1—O1	90.98 (16)	O3—Cu2—N7	162.1 (2)
O5—Cu1—N3	92.07 (18)	O3—Cu2—N5	90.4 (2)

O5—Cu1—N1	159.71 (19)	O7—Cu2—O3	90.19 (18)
N3—Cu1—O1	165.27 (17)	O7—Cu2—N7	90.48 (19)
N1—Cu1—O1	89.47 (17)	O7—Cu2—N5	163.3 (2)
N1—Cu1—N3	92.62 (19)	N7—Cu2—N5	94.1 (2)

Hydrogen bond geometry (Å, o)

D–H···A	D–H	H···A	D···A	D–H···A
O11—H11A···O10	0.85	1.91	2.753 (7)	173
O11—H11B···O5	0.85	1.88	2.729 (6)	173
N4—H4···O1^{i}	0.86	2.13	2.930 (6)	155
N2—H2···O11^{ii}	0.86	2.01	2.851 (7)	167
O10—H10C···O9	0.85	1.96	2.807 (8)	174
O10—H10D···O2^{iii}	0.85	1.96	2.803 (6)	172
N6—H6···O12^{iv}	0.86	1.96	2.804 (8)	167
O12—H12D···O4	0.85	2.17	2.890 (7)	142
O12—H12E···O7	0.85	1.91	2.732 (7)	162
N8—H8···O13	0.86	1.96	2.780 (7)	160
O9—H9C···O13^{v}	0.85	2.03	2.881 (8)	174

*Symmetry codes: (i) *x*−1/2, *y*, −*z*+3/2; (ii) *x*+1/2, *y*, −*z*+3/2; (iii) −*x*+1/2, *y*+1/2, *z*; (iv) *x*−1/2, −*y*+3/2, −*z*+1; (v) −*x*+1, −*y*+2, −*z*+1; (vi) *x*+1/2, −*y*+3/2, −*z*+1.

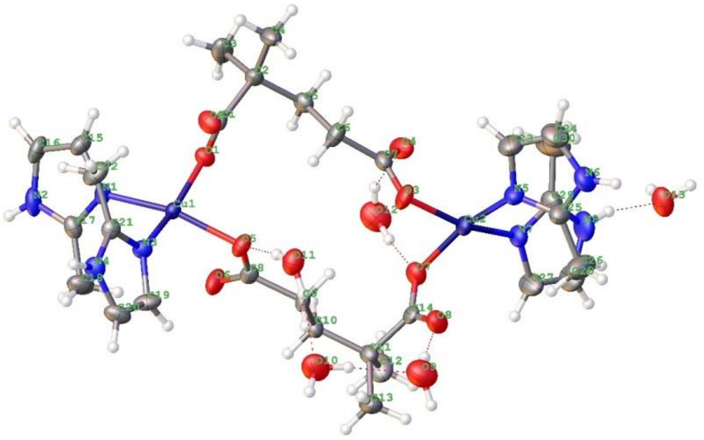


Figure 1. The molecular structure of **1.**

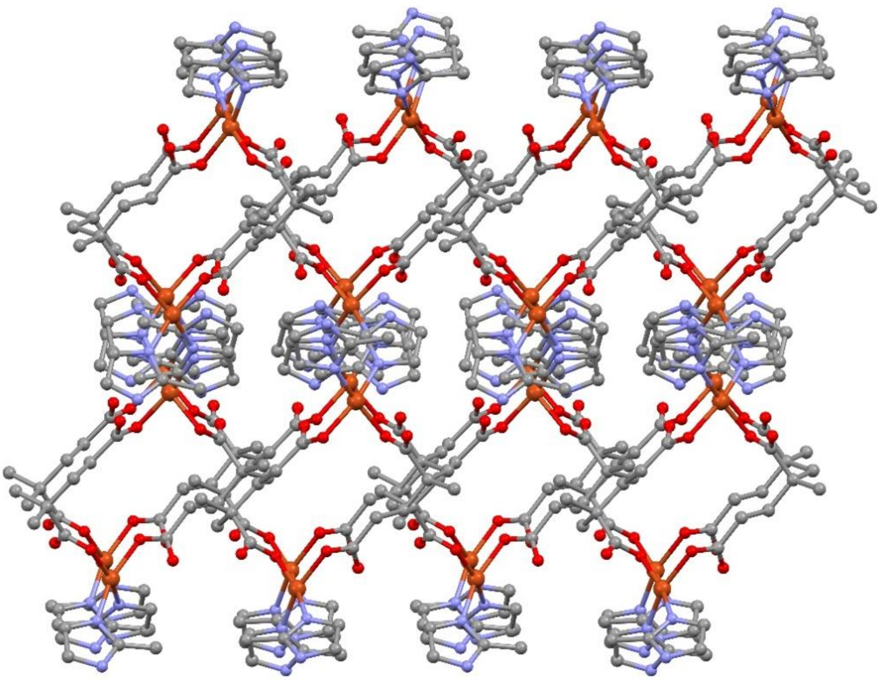


Figure 2: 3D supramolecular structure of complex **1** generated by hydrogen bonds and C–H···π, C–O···π and π···π interactions.

{[Cd(μ-dmg)(2-meim)$_2$]·H$_2$O}$_n$ (2). The molecular structure of complex **2** consists of polymeric [Cd(μ-dmg)(2-meim)$_2$] units (Figure 3). The complex has the monoclinic crystal system with the *P*21/n space group (Table 2.). The asymmetric unit consists of one Cd(II) ion, one dmg ligand, two 2-meim ligands and one water molecule (Scheme 1). As shown in Figure 3, Cd ion has distorted octahedral geometry and linked to four carboxylate oxygens from two different dmg ligands and two nitrogen atoms from 2-meim ligands (CdO_4N_2). The Cd(II) ions in **2** are bound by dmg ligands to form 1D zig-zag chains. The adjacent 1D chains are linked with the dmg to form the zig-zag-like structure shown in Figure 4. The neighboring 1D chains are expanded into 2D supramolecular layer with hydrogen bonding between carboxylate oxygen atom (O1) of dmg and uncoordinated water molecule (O5) [O1···O5 = 2.85 Å, O1···H5D-O5 = 170.06°] and between the imidazole nitrogen atom (N4) of adjacent 2-meim ligand and water molecule (O5) [N4···O5 = 2.69 Å, N4-H4···O5 = 166.89°] (Figure 5). Furthermore, 2D supramolecular structures form a three dimensional (3D) supramolecular network through C–H···π and C–O···π interactions. (Figure 6).

Table 3. Selected bond distances (Å), angles (°) and hydrogen-bond geometry data for **2***.

Bond Lengths (Å)			
Cd1—O1	2.344 (6)	Cd1—N3	2.241 (7)
Cd1—O2	2.415 (7)	Cd1—O3^{i}	2.375 (7)
Cd1—N1	2.225 (8)	Cd1—C7^{i}	2.714 (9)
Cd1—O4^{i}	2.356 (8)	O3—Cd1^{ii}	2.375 (7)
Angles (°)			
O1—Cd1—O2	54.4 (2)	O4^{i}—Cd1—O2	87.0 (3)
O1—Cd1—O4^{i}	97.2 (3)	O4^{i}—Cd1—O3^{i}	54.2 (3)
O1—Cd1—O3^{i}	147.2 (3)	O4^{i}—Cd1—C7^{i}	27.1 (3)
O1—Cd1—C7^{i}	122.3 (3)	N3—Cd1—O1	108.0 (3)
O2—Cd1—C7^{i}	94.2 (3)	N3—Cd1—O2	88.1 (3)
N1—Cd1—O1	97.0 (3)	N3—Cd1—O4^{i}	144.7 (3)
N1—Cd1—O2	151.3 (2)	N3—Cd1—O3^{i}	93.3 (3)
N1—Cd1—O4^{i}	98.5 (3)	N3—Cd1—C7^{i}	119.0 (3)

N1—Cd1—N3	102.3 (3)	O3^{i}—Cd1—O2	103.4 (3)
N1—Cd1—O3^{i}	102.6 (3)	O3^{i}—Cd1—C7^{i}	27.2 (3)
N1—Cd1—C7^{i}	103.5 (3)		

Hydrogen bond geometry (Å, o)

D–H···A	D–H	H···A	D···A	D–H···A
N2—H2···O2^{iii}	0.86	1.89	2.731 (11)	165
N4—H4···O5	0.86	1.85	2.693 (12)	167
O5—H5C···O3^{iv}	0.85	1.86	2.714 (11)	179
O5—H5D···O1^{v}	0.85	2.02	2.857 (11)	170

*Symmetry codes: (iii) x+1/2, −y+1/2, z+1/2; (iv) x+1/2, −y+1/2, z−1/2; (v) −x+3/2, y−1/2, −z+1/2.

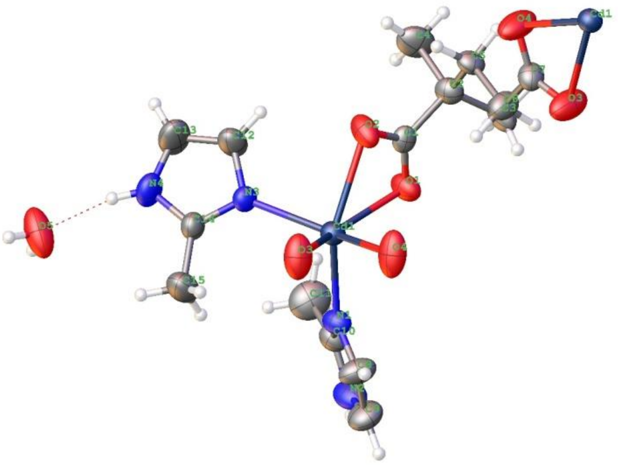


Figure 3: The molecular structure of **2.**

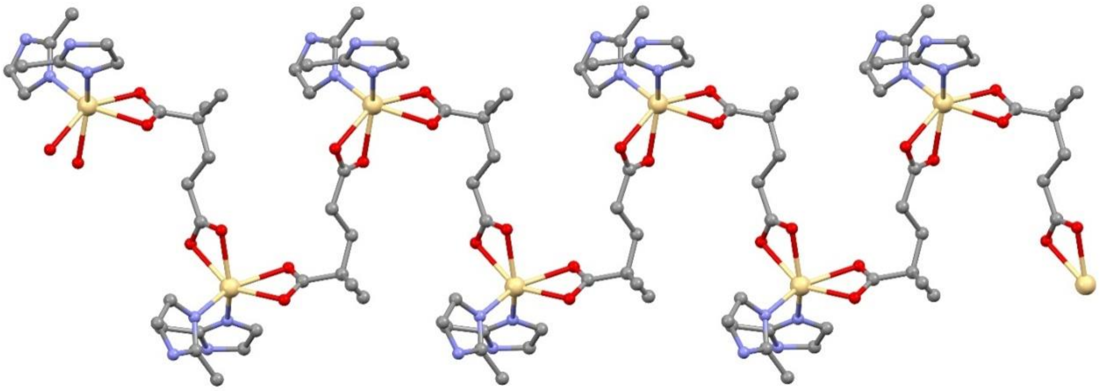


Figure 4: 1D zig-zag polymeric structure of **2.**

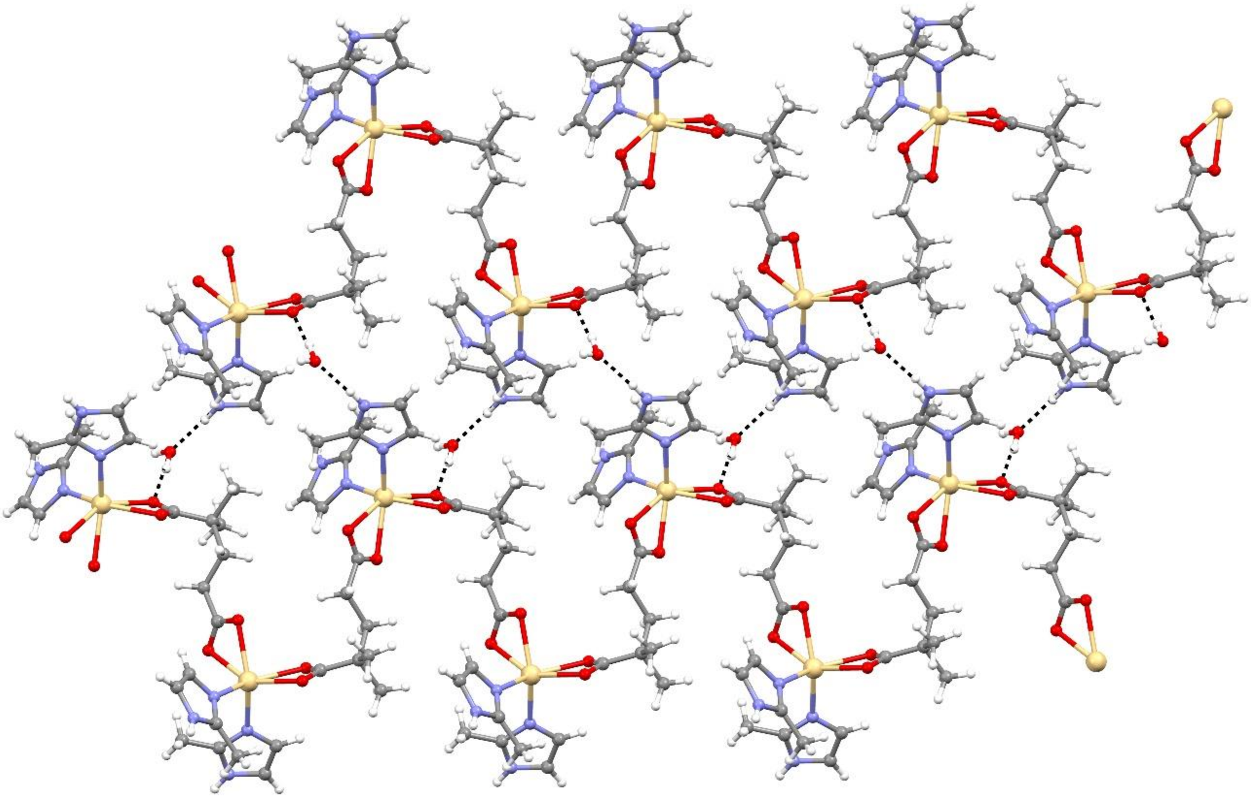


Figure 5: 2D supramolecular layer of complex **2** generated by hydrogen bondings.

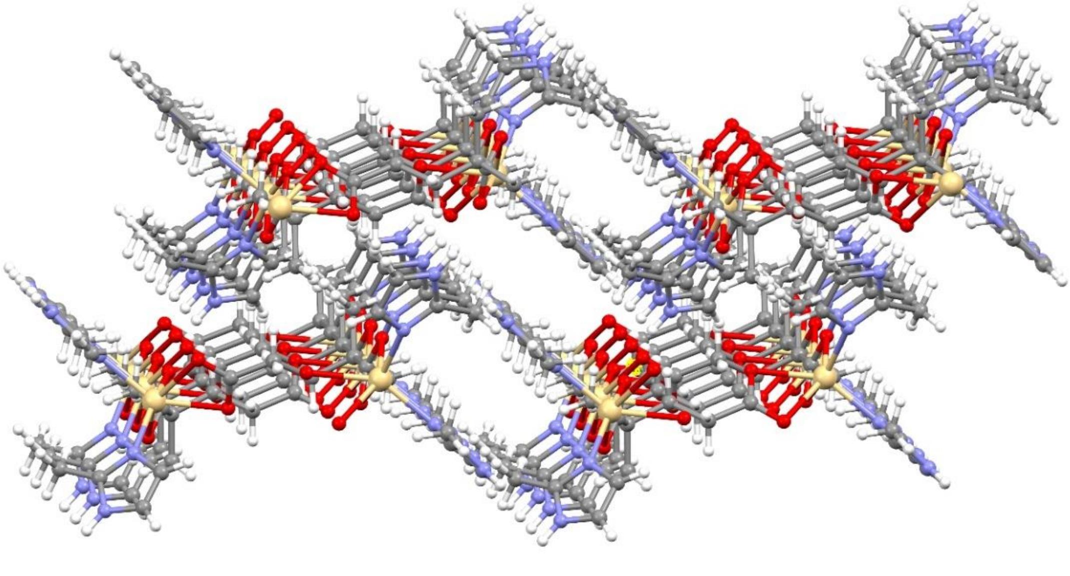


Figure 6: 3D supramolecular structure of complex **2** generated by C–H···π and C–O···π interactions.

Powder X-ray diffraction (PXRD) and Thermal Analyses of complexes 1 and 2

The complexes of the PXRD patterns obtained from the crystal structures are compatible with the simulated patterns obtained from single crystal results; indicating the phase purity of the complexes **1** and **2** (Figure 7 and 8).

TG, DTG and DTA curves for complexes 1 and 2 were obtained in static air atmospheres between 30-1000 °C (Figure 9 and 10). For complex 1, three stage thermal decomposition is observed. The first stage starts at about 60 °C with the release of the five water molecules and ends at 104 °C (found 10.18%; theoretical value 10.44%). The diversion of water is at low temperature because the water molecule is not coordinated as shown by the crystal structure (Figure 7). The second step occurs between 284 and 334 °C which corresponds to the release of one 2-meim ligand and one dmg ligand (found 57.69%; theoretical value 56.79%). The following stage between 379 and 457 °C involves the decay of one dmg molecule. Also the highest exothermic mass loss occurs at this step. The final decomposition product is 2 molecules of CuO (found: 17.81%; theoretical value 18.33%). The Cd complex is stable up to 112 °C. The first stage between 112 and 138 °C corresponds to the endothermic removal of uncoordinated water molecule (found 4.10; theoretical value 3.90%). The second weight loss takes place between 226 and 271 °C, corresponding to the decomposition of one 2-meim ligand (found 8.73%; theoretical value 9.07%). The remaining organic ligands are degradation between 415 and 506 °C (found 52.77%; theoretical value 53.55%) by the most severe exothermic effect (DTA_{max} = 460 °C). Total mass loss is 73.22% (theoretical value 71.60%) as a result of the thermal degradation of complex 2 (Figure 8). The final product for the complex 2 is suggested to be CdO.

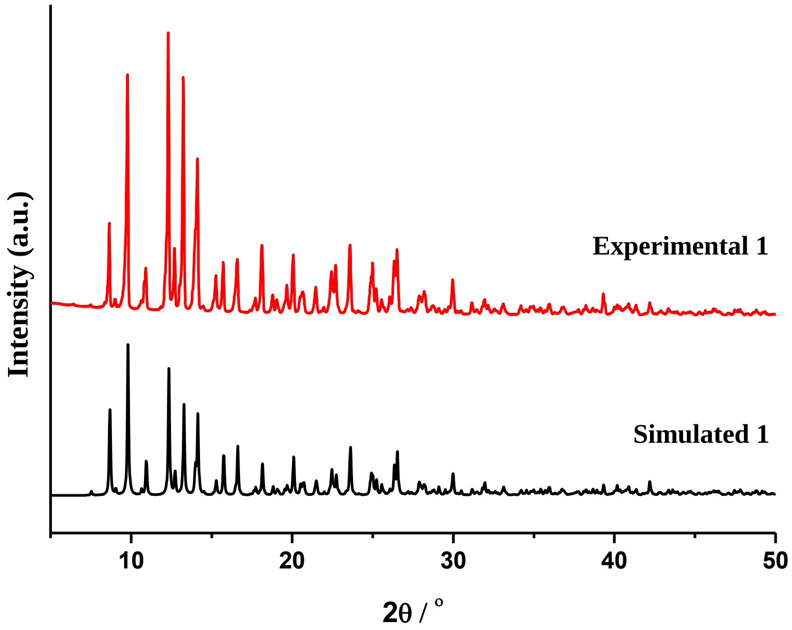


Figure 7 PXRD pattern of $[Cu_2(\mu\text{-dmg})_2(2\text{-meim})_4]_2.5H_2O$ **(1).**

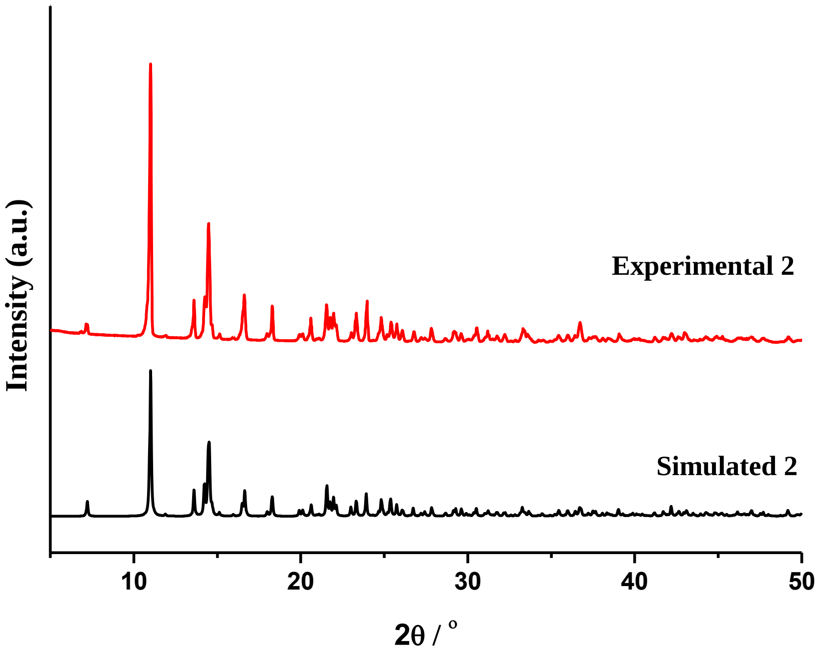


Figure 8 PXRD pattern of $\{[Cd(\mu\text{-dmg})(2\text{-meim})_2]\cdot H_2O\}_n$ **(2).**

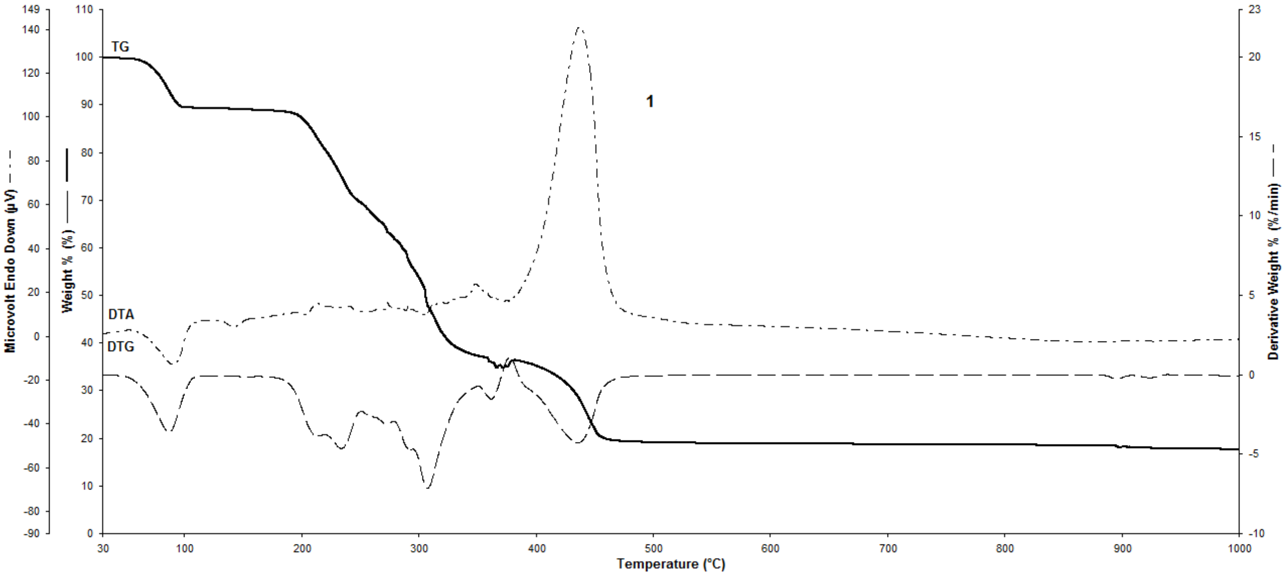


Figure 9. Thermal analysis curves of $[Cu_2(\mu\text{-dmg})_2(2\text{-meim})_4]_2 \cdot 5H_2O$ **(1).**

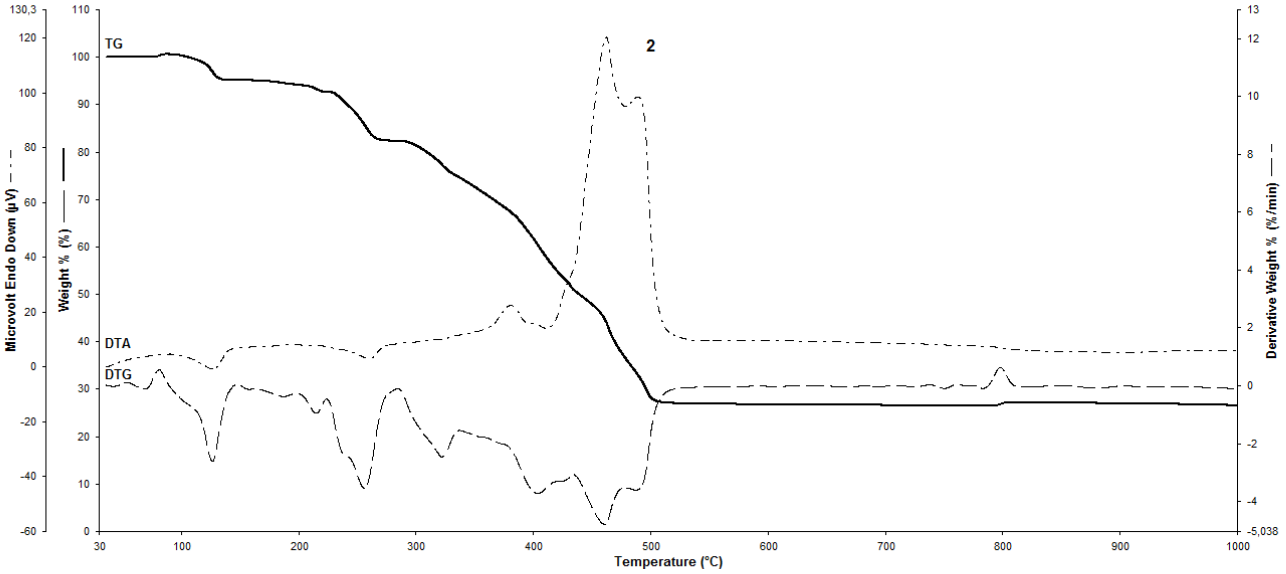


Figure 10. Thermal analysis curves of $\{[Cd(\mu\text{-dmg})(2\text{-meim})_2]\cdot H_2O\}_n$ **(2).**

CONCLUSION

In summary, new dimeric Cu(II) and polymeric Cd(II) coordination compounds with 2-meim and flexible dmg ligands have been successfully synthesized and characterized by spectroscopic and thermal studies. The dmg ligand in all of the complexes coordinated to the metal centers as a bidentate fashion and acted as a bridging ligand between the metal(II) centers to form dimeric $[Cu_2(\mu\text{-dmg})_2]$ units for complex **1** and 1D zig-zag polymeric structure for complex **2**. The reason for the difference may be due to the radius variations of the metal (II) ions. In addition, the Cd(II) ion may have more coordination ability than the Cu(II) ion. Nevertheless, more studies are necessary to support or disprove such a claim. The compounds decompose in three main thermal stages. The final decomposition products corresponds to the metal oxides.

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