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Thermal properties of solid complexes with biologically important heterocyclic ligands

Part IV. Thermal and spectral properties of 2-chloro- and 2-bromobenzoato Cu(II) complexes with nicotinamide and different bonded water molecules

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Abstract Thermogravimetry (TG), derivative thermogravimetry (DTG) and infrared (IR) spectroscopy have been applied to the investigation of the thermal behaviour and structure of the compounds $[Cu(2\text{-}Clbz)_2(nia)_2(H_2O)_2]$ (I), $[Cu(2\text{-}Clbz)_2(nia)_2]\cdot H_2O$ (II), $[Cu(2\text{-}Brbz)_2(nia)_2]\cdot 2H_2O$ (III), $[Cu(2\text{-}Brbz)_2(nia)_2(H_2O)]$ (IV), where 2-Clbz and 2-Brbz = 2-chloro- and 2-bromobenzoate anions, nia = nicotinamide, H_2O = water molecules. Thermal decomposition of all studied compounds proceeds in three steps. Heating the compounds first results in a release of non-coordinated and/or coordinated water molecules. The final product of thermal decomposition was CuO. The thermal stability of the complexes can be ordered in the sequence: I<IV<III<II. Nicotinamide is coordinated to Cu(II) through the nitrogen atom of the heterocyclic ring. IR data suggest the unidentate coordination of benzoate anions to Cu(II) in complexes I, IV and bidentate coordination in complexes II and III.

Keywords TG · DTG · IR spectra · Heterocyclic compounds · Cu(II) complexes

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Introduction

Heterocyclic compounds play a significant role in many biological systems, especially N-donor ligand systems being a component of several vitamin and drugs [1–3]. Nicotinamide is a nitrogen-donor ligand that has been used in the treatment of various skin diseases (atopic eczema [4], psoriasis and skin cancer [5]). In order to enhance understanding of drug–metal ion interactions, we have been studying the thermal and spectral properties of Cu (II) complexes with 2-chloro- and 2-bromobenzoic acid (2-ClbzH, 2-BrbzH) and nicotinamide (nia) (Fig. 1).

The revealing information on relationship between the structure and thermolysis of metal complexes, and the study of metal and ligand nature on the process of thermal decomposition are very important. This study is a continuation of previously reported studies [6–9] on the thermal and spectral properties of metal complexes with pyridine derivatives. Thermal and spectral analyses are very useful methods for material's characterization. Therefore, many authors have applied these techniques for various material's characterization [10–30]. This paper describes the preparation of complexes: $[Cu(2\text{-}Clbz)_2(nia)_2(H_2O)_2]$ (I), $[Cu(2\text{-}Clbz)_2(nia)_2]\cdot H_2O$ (II), $[Cu(2\text{-}Brbz)_2(nia)_2]\cdot 2H_2O$ (III) and $[Cu(2\text{-}Brbz)_2(nia)_2(H_2O)_2]$ (IV) with different bonded water molecules [31, 32], along with thermal and spectral (IR) analyses of prepared compounds.

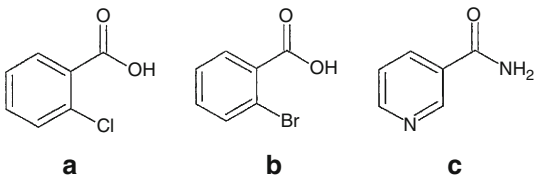

Fig. 1 Structures of **a** 2-ClbzH, **b** 2-BrbzH and **c** nia

Experimental

Preparation of compounds

[Cu(2-Clbz)$_2$(nia)$_2$(H$_2$O)$_2$] (I) (molar ratio Cu(Ac)$_2$: nia:2-ClbzH is 1:2:2)

The complex I was prepared by treating nicotinamide (2 mmoL, 244 mg) with copper acetate [Cu(ac)$_2$, 1 mmoL, 200 mg, 10 mL] in water (90 mL). The solution was stirred until nicotinamide was dissolved. Then to the solution was added 2-chlorobenzoic acid (2 mmoL, 313 mg). Then solution was filtrered off and reduced to room temperature to crystallize.

[Cu(2-Clbz)$_2$(nia)$_2$]·H$_2$O(II) (molar ratio Cu(Ac)$_2$: nia:2-ClbzH is 1:4:2)

The complex II was prepared by treating nicotinamide (4 mmoL, 488 mg) with copper acetate [Cu(ac)$_2$, 1 mmoL, 200 mg, 10 mL] in water (15 mL). The solution was stirred until nicotinamide was dissolved. Then 2-chlorobenzoic acid (2 mmoL, 313 mg) was added to the solution the resulting solution was filtrered off and reduced to room temperature to crystallize.

[Cu(2-Brbz)$_2$(nia)$_2$]·2H$_2$O (III) (molar ratio Cu(Ac)$_2$:nia:2-BrbzH is 1:2:2)

The complex III was prepared by treating nicotinamide (2 mmoL, 244 mg) with copper acetate [Cu(ac)$_2$, 1 mmoL, 200 mg, 10 mL] in water (15 mL). The solution was stirred until nicotinamide was dissolved. Then 2-bromobenzoic acid (2 mmoL, 402 mg) was added to the solution and the resulting solution was stirred with a magnetic stirrer for several days. Then solution was filtrered off and reduced to room temperature to crystallize.

[Cu(2-Brbz)$_2$(nia)$_2$(H$_2$O)] (IV) (molar ratio Cu(Ac)$_2$: nia:2-BrbzH is 1:2:2)

The complex IV was prepared by treating nicotinamide (2 mmoL, 244 mg) with copper acetate [Cu(ac)$_2$, 1 mmoL, 200 mg, 10 mL] in water (90 mL). The solution was stirred until nicotinamide were dissolved. Then to the solution was added 2-bromobenzoic acid (2 mmoL, 402 mg). The solution was stirred with a magnetic stirrer for several days. Then solution was filtrered off and reduced to room temperature to crystallize.

Measurements

Elemental analyses (C, H, and N) were carried by means of Carlo Erba 1106 Analyser. The infrared (IR) spectra were obtained on Philips analytical PU 9800 FTIR spectrometer in the range 400–4,000 cm^{-1}.

Thermal decomposition studies were carried out on a Paulik–Paulik–Erdes Derivatograph (Type 00102, MOM Budapest) in air atmosphere using a platinum crucible with a sample mass of 150 mg. 10 °C min^{-1} was chosen as the rate of temperature increase of for all the measurements.

Results and discussion

Analytical results of studied compound

The contents of C, H, and N were determined by elemental analysis, and the content of Cu (II) was established by complexometric titration. The analytical data of the compounds I–IV reported in Table 1 shows a good agreement between the experimental and calculated data.

Thermal decomposition of studied compounds

The thermal decomposition data of the compounds I–IV are shown in Table 2. Thermal decompositions the studied compounds are multi-stage processes. The subsequent

Table 1 Elemental analyses and complexometric titration data of the complexes I–IV

Complex	Theoretical/%				Experimental/%			
	C	H	N	Cu	C	H	N	Cu
[Cu(2-Clbz)$_2$(nia)$_2$(H$_2$O)$_2$] (I)	47.68	3.69	8.55	9.70	47.70	3.67	8.53	9.69
[Cu(2-Clbz)$_2$(nia)$_2$]·H$_2$O (II)	47.68	3.69	8.55	9.70	47.72	3.66	8.51	9.72
[Cu(2-Brbz)$_2$(nia)$_2$]·2H$_2$O (III)	41.98	3.25	7.53	8.54	42.22	3.22	7.56	8.52
[Cu(2-Brbz)$_2$(nia)$_2$ (H$_2$O)$_2$] (IV)	41.98	3.25	7.53	8.54	42.24	3.23	7.61	8.51

Table 2 Thermal decomposition data of the complexes I–IV

Complex	DTG		TG	
	T_{max}/°C	Temp. interval/°C	Δ*m*(found/calc.)/%	Lost component
I	69	42–134	5.50/5.50	2 H_2O
	219	134–420	61.00/61.04	$(nia)_2$+(2-Clbz)
	568	420–660	24.00/23.75	(2-Clbz)
II	96	48–139	3.00/2.83	H_2O
	219	139–419	63.00/62.77	$(nia)_2$+(2-Clbz)
	603	419–681	25.00/24.42	(2-Clbz)
III	83	43–141	5.00/4.84	2 H_2O
	218	141–426	60.00/59.71	$(nia)_2$+(2-Brbz)
	607	426–738	27.00/26.89	(2-Brbz)
IV	80	46–141	5.00/4.84	2 H_2O
	219	141–442	59.50/59.71	$(nia)_2$+(2-Brbz)
	618	442–729	26.50/26.89	(2-Brbz)

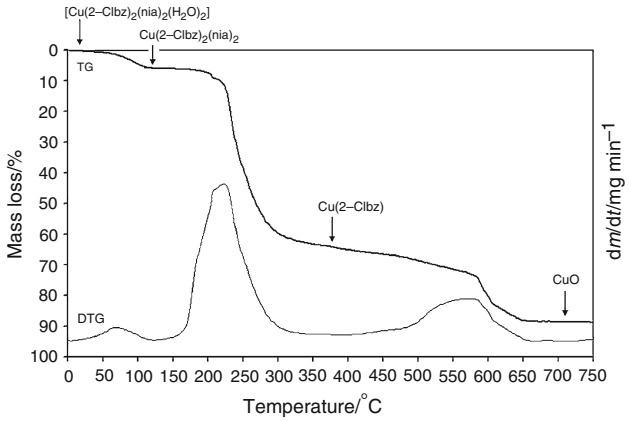

Fig. 2 TG and DTG curves of $[Cu(2\text{-}Clbz)_2(nia)_2(H_2O)_2]$ (I)

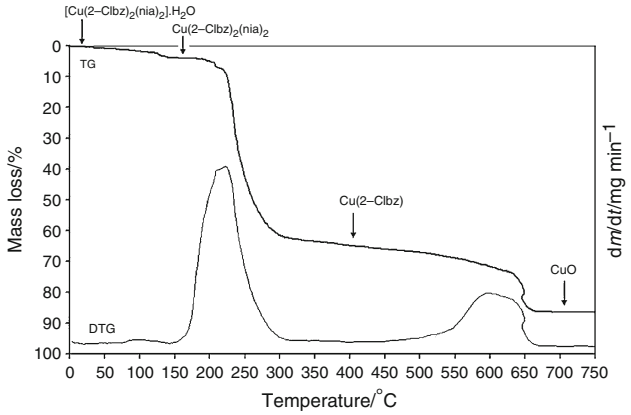

Fig. 3 TG and DTG curves of $[Cu(2\text{-}Clbz)_2(nia)_2]\cdot H_2O$ (II)

detachment of the ligands was observed. The final solid product was always identified as CuO.

The TG and DTG curves for $[Cu(2\text{-}Clbz)_2(nia)_2(H_2O)_2]$ (I) are shown in Fig. 2. The TG curve of this complex indicates that it is thermally stable up to ~42 °C, when the successive decomposition to CuO begins, the final product is formed at ~660 °C. This is followed by three mass-loss steps of ~42–134, 134–420 and 420–460 °C. Based on these mass-loss values (Table 2), these three steps were attributed to the formation of two intermediate products, i.e. $Cu(2\text{-}Clbz)_2(nia)_2$, and Cu(2-Clbz), and final product, CuO. The thermal decomposition scheme is as follows:

$$\left[Cu(2\text{-}Clbz)_2(nia)_2(H_2O)_2\right] \xrightarrow{42-134\,^{\circ}C} Cu(2\text{-}Clbz)_2(nia)_2 \quad (1)$$

$$Cu(2\text{-}Clbz)_2(nia)_2 \xrightarrow{134-420\,^{\circ}C} Cu(2\text{-}Clbz) \quad (2)$$

$$Cu(2\text{-}Clbz) \xrightarrow{420-660\,^{\circ}C} CuO \quad (3)$$

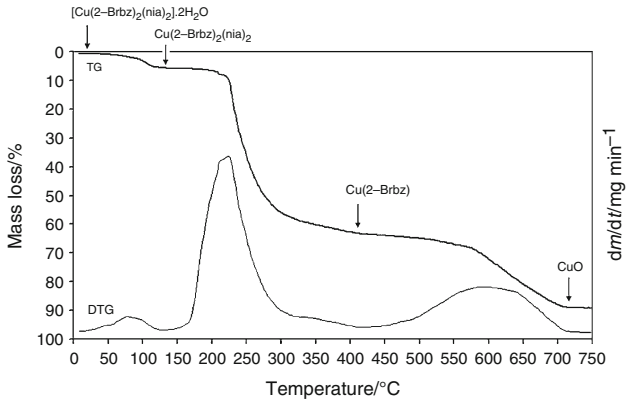

Fig. 4 TG and DTG curves of $[Cu(2\text{-}Brbz)_2(nia)_2]\cdot 2H_2O$ (III)

The DTG curve for complex I (Fig. 2) presents three maxima at ~69, 219 and 568 °C corresponding to the losses of 2 H_2O, 2 nia+2-Clbz and 2-Clbz, respectively (Table 2).

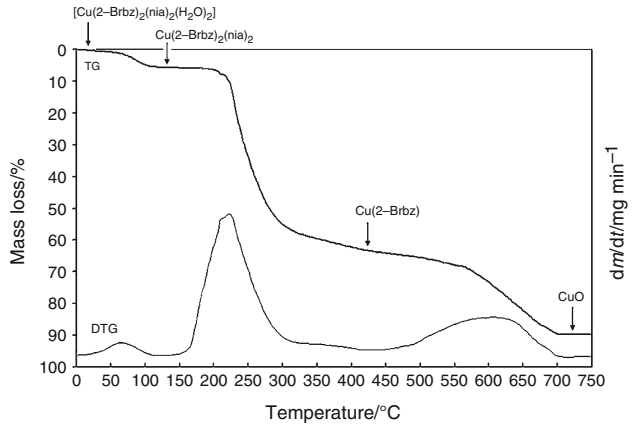

Fig. 5 TG and DTG curves of $[Cu(2\text{-}Brbz)_2(nia)_2(H_2O)_2]$ (IV)

Table 3 Important infrared bands (in cm^{-1}) of complexes I–IV

Assignment	Complex			
	I	II	III	IV
ν(OH)	3,550	3,550	3,384	3,554
$\nu(NH_2)$(as)	3,341	3,340	3,304	3,375
$\nu(NH_2)$(s)	3,196	3,194	3,196	3,205
ν(CO)	1,696	1,670	1,693	1,696
δ(HOH)	1,630	1,630	1,620	1,623
ν(CO)	1,607	1,598	1,607	1,598
$\nu(COO^-)$(as)	1,637	1,580	1,571	1,643
$\nu(COO^-)$(s)	1,446	1,428	1,429	1,429
$\Delta\nu(COO^-)$	191	152	142	214

The TG and DTG curves for $[Cu(2\text{-}Clbz)_2(nia)_2]\cdot H_2O$ (II) are shown in Fig. 3. The TG curve for this complex indicates that it is thermally stable up to ~48 °C, where the dehydration process commences. This is followed by three mass-loss steps of ~48–139, 139–419 and 419–681 °C. Based on these mass-loss values (Table 2), these three steps were attributed to the formation of two intermediate decomposition products, i.e. $Cu(2\text{-}Clbz)_2(nia)_2$ and Cu(2-Clbz), while the final solid product is concluded to be CuO. The most probable thermal decomposition scheme is as follows:

$$[Cu(2\text{-}Clbz)_2(nia)_2]\cdot H_2O \xrightarrow{48-139\,^\circ C} Cu(2\text{-}Clbz)_2(nia)_2 \quad (4)$$

$$Cu(2\text{-}Clbz)_2(nia)_2 \xrightarrow{139-419\,^\circ C} Cu(2\text{-}Clbz) \quad (5)$$

$$Cu(2\text{-}Clbz) \xrightarrow{419-681\,^\circ C} CuO \quad (6)$$

The DTG curve for the complex II (Fig. 3) displays three maxima of peaks at ~96, 219 and 603 °C, corresponding to the losses of H_2O, 2 nia+2-Clbz and 2-Clbz, respectively (Table 2).

The TG and DTG curves for $[Cu(2\text{-}Brbz)_2(nia)_2]\cdot 2H_2O$ (III) are shown in Fig. 4. The TG curve for this complex indicates that it is thermally stable up to ~43 °C, where the dehydration process commences. This is followed by three mass-loss steps of ~43–141, 141–426 and 426–738 °C. Based on these mass-loss values (Table 2), these three steps were attributed to the formation of two intermediate decomposition products, i.e. $Cu(2\text{-}Brbz)_2(nia)_2$ and Cu(2-Brbz), while the final solid product is concluded to be CuO. The most probable thermal decomposition scheme is as follows:

$$[Cu(2\text{-}Brbz)_2(nia)_2]\cdot 2H_2O \xrightarrow{43-141\,^\circ C} Cu(2\text{-}Brbz)_2(nia)_2 \quad (7)$$

$$Cu(2\text{-}Brbz)_2(nia)_2 \xrightarrow{141-426\,^\circ C} Cu(2\text{-}Brbz) \quad (8)$$

$$Cu(2\text{-}Brbz) \xrightarrow{426-738\,^\circ C} CuO \quad (9)$$

The DTG curve for complex III (Fig. 4) presents three maxima of peaks at ~83, 218 and 607 °C corresponding to the losses of 2 H_2O, 2 nia+2-Brbz and 2-Brbz, respectively (Table 2).

The TG and DTG curves for $[Cu(2\text{–}Brbz)_2(nia)_2(H_2O)_2]$ (IV) are shown in the Fig. 5. The TG curve for this complex indicates that it is thermally stable up to ~46 °C, when the decomposition to CuO commences, and the final product formed at ~729 °C. The TG curve shows three bendings at ~46, 141 and 442 °C. These correspond to the presence of two intermediate decomposition products: Cu(2-Brbz) $(nia)_2$ and Cu(2-Brbz), while the final solid

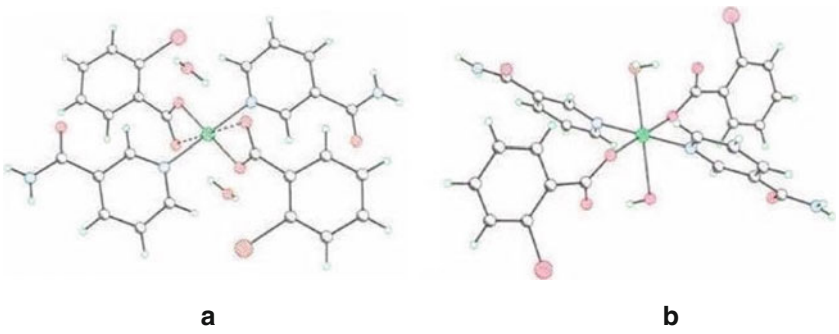

Fig. 6 Molecular structure of complexes **a** $[Cu(2\text{-}Brbz)_2(nia)_2]\cdot 2H_2O$, **b** $[Cu(2\text{-}Brbz)_2(nia)_2(H_2O)_2]$

product is concluded to be CuO. The thermal decomposition scheme is as follows:

$$\left[\mathrm{Cu(2\text{-}Brbz)_2(nia)_2(H_2O)_2}\right] \xrightarrow{46-141\,^{\circ}\mathrm{C}} \mathrm{Cu(2\text{-}Brbz)_2(nia)_2} \quad (10)$$

$$\mathrm{Cu(2\text{-}Brbz)_2(nia)_2} \xrightarrow{141-442\,^{\circ}\mathrm{C}} \mathrm{Cu(2\text{-}Brbz)} \quad (11)$$

$$\mathrm{Cu(2\text{-}Brbz)} \xrightarrow{442-729\,^{\circ}\mathrm{C}} \mathrm{CuO} \quad (12)$$

The DTG curve for complex IV (Fig. 5) presents three maxima of peaks at ~80, 219 and 618 °C, corresponding to the losses of 2 H_2O, 2 nia + 2-Brbz and 2-Brbz, respectively (Table 2).

IR spectra

The modes of the coordinated ligands in the studied complexes have been investigated by means of IR absorption spectra. Spectral results have been correlated with structural data obtained by means of X-ray analysis [31, 32]. The most important IR frequencies attributed to the vibrations of the complexes I–IV are reported in Table 3.

The IR spectra of complexes I–IV show broad absorption bands in the range 3,384 –3,550 cm^{-1}. These frequencies correspond to the asymmetric and symmetric OH stretching [1, 33]. These bands clearly confirm the presence of water in the complexes I–IV.

The compounds showed the carboxylate stretching frequencies $\nu(COO^-)$(s) in the range 1,428–1,446 cm^{-1} and $\nu(COO^-)$(as) in the range 1,670–1,696 cm^{-1} (Table 3). The position of the bands is characteristic of metal(II) carboxylate compounds [33]. Carboxylate ions can coordinate to metal ions in a number of ways such as unidentate, bidentate (chelating) or bridging, and there is an evidence of this fact in the IR spectrum. The analysis of COO^- group bands frequencies allowed for the determination of the parameter $\Delta\nu(COO^-) = \nu(COO^-)(as) - \nu(COO^-)(s)$. The magnitude of $\Delta\nu(COO^-)$ has been used by Nakamoto [33] as a criterion for the way carboxylate binds with metal ions. Calculated from the examined spectra, values of $\Delta\nu(COO^-)$ for complexes I, and IV in the range 191–214 cm^{-1} and for complexes II, and III in the range 142–152 cm^{-1}. These values are in good accordance with the literature data for unidentate-bonded benzoate anions in complexes I, and IV and bidentate bonded benzoate anions in complexes II, and III. The stretching vibration of the C=H in the pyridine ring of the nicotinamide appeared at 1,590 cm^{-1} [34]. Upon complex formation, the peak shifts to higher frequencies [35]. The shifts in complexes I–IV (in the range 1,597–1,607 cm^{-1}) may suggest that bond formation on the metal with the N of pyridine ring.

The obtained conclusions from the spectral analysis correlate with the results of X-ray analysis [31, 32]. The structures of $[Cu(2\text{-}Brbz)_2(nia)_2]\cdot 2H_2O$ and $[Cu(2\text{-}Brbz)_2(nia)_2(H_2O)_2]$ are represented in Fig. 6 as examples.

Conclusions

All of the complexes I–IV are hydrated; however, water molecules are coordinated to Cu(II)in the complexes I and IV. In all the studied complexes, losses of decomposition products occur (on the TG and DTG curves) in three steps. The coordinated and/or noncoordinated water molecules have no influence on the stoichiometry of thermal decomposition of the studied complexes, but thermal stability (on the temperature of the first DTG peak) is changed. The thermal stability of the complexes can be ordered in the sequence: I<IV<III<II.The results reveal that CuO is left as a residue at the end of the thermal degradation experiments of the compounds I–IV.

IR data are in good accordance with the literature data and X-ray analysis results for unidentate-bonded benzoate anions in complexes I and IV and bidentately bonded in complexes II and III. Nicotinamide (nia) is coordinated to Cu(II) through the nitrogen atom of heterocyclic ring in the studied complexes.

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