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Synthesis, characterization and thermal behaviour of solid-state 3-methoxybenzoates of heavy trivalent lanthanides and yttrium(III)

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Abstract Solid-state $Ln(L)_3$ compounds, where Ln stands for trivalent Tb, Dy, Ho, Er, Tm, Yb, Lu and Y, and L is 3-methoxybenzoate, have been synthesized. X-ray powder diffractometry, infrared spectroscopy, complexometry and elemental analysis were used to characterize the compounds. In order to study the thermal behaviour of these compounds simultaneous thermogravimetry and differential thermal analysis (TG-DTA) and differential scanning calorimetry (DSC) were used. The results provided information on the composition, dehydration, polymorphic transformation, thermal stability and thermal decomposition of the synthesized compounds.

Keywords Characterization · Heavy lanthanides · 3-methoxybenzoate · Thermal behaviour

Introduction

The literature shows that several metal–ion compounds of benzoate, C_6H_5–COO^- (Bz), and phenyl-substituted derivatives of benzoate have been investigated in aqueous solutions [1–3] and in solid state [4–18]. Benzoic acid and some of their derivatives have been used as conservant, catalyst precursors, in pharmaceutical industries, beyond other applications.

In aqueous solutions, the studies reported the thermodynamics of complexation; spectroscopy study; influence of pH; surfactant and synergic agent on the luminescent properties; crystal structure, photophysical and magnetic properties [1–4].

In the solid state, the studies reported the thermal and spectral behaviour of benzoates and its derivatives with rare earth elements [5, 6, 8, 12–14, 16–19]; the vibrational and electronic spectroscopy study of lanthanides and effect of sodium on the aromatic system of benzoic acid [20, 21]; the reaction of divalent copper, cobalt and nickel with 3-hydroxy-4-methoxy and 3-methoxy-4-hydroxybenzoic acid and a structure for these compounds has been proposed on the basis of spectroscopy and thermogravimetric data [22]. The thermal decomposition of thorium salts of benzoic and 4-methoxybenzoic acids in air atmosphere [23] and thermal behaviour of solid state 4-methoxy; 3-methoxy; 2-methoxybenzoates and benzoate of some bivalent transition and trivalent metal ions have also been described [9–17].

In this study, 3-methoxybenzoates of trivalent Tb, Dy, Ho, Er, Tm, Yb, Lu and Y were synthesized and characterized by means of X-ray powder diffractometry, infrared spectroscopy, thermogravimetry and differential thermal analysis (TG-DTA), differential scanning calorimetry (DSC) and complexometry. This article is a continuation of the work of references [16, 17].

Experimental

The 3-methoxybenzoic acid (3-MeO-HBz) with 99% purity was obtained from Aldrich. Aqueous solution of Na-3-MeO-Bz 0.1 mol L^{-1} was prepared from aqueous 3-MeO-HBz suspension by adding sodium hydroxide solution

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2.5 mol L^{-1} up to near total neutralization, followed by NaOH solution 0.1 mol L^{-1} up to pH = 7.9.

Lanthanide chlorides were prepared from the corresponding metal oxides by treatment with concentrated hydrochloric acid. The resulting solutions were evaporated to near dryness; the residues were again dissolved in distilled water, transferred to a volumetric flask and diluted to obtain ca. 0.1 mol L^{-1} solutions, whose pH was adjusted to 5.0 by adding diluted sodium hydroxide or hydrochloric acid solutions.

The solid-state compounds were prepared by adding slowly, with continuous stirring, the solution of the ligand to the respective metal chloride until total precipitation of the metal ions. The precipitates were washed with distilled water until elimination of chloride ions (qualitative test with $AgNO_3/HNO_3$ solution), filtered through, dried on Whatman no 42 filter paper and kept in a desiccator over anhydrous calcium chloride.

In the solid-state compounds, hydration water, ligand and metal ion content were determined from TG curves. The metal ions were also determined by complexometric titrations with standard EDTA solution using xylenol orange as indicator [24].

X-ray powder patterns were obtained by using a SIEMENS D-5000 X-ray diffractometer employing CuK_α radiation ($\lambda = 1.541$ Å) and setting of 40 kV and 20 mA.

Infrared spectra for Na-3-MeO-Bz as well as for its metal ion compounds were run on a Nicolet model Impact 400 FT-IR Instrument, within the 4000–400 cm^{-1} range. The solid samples were pressed into KBr pellets.

Simultaneous TG-DTA and DSC curves were obtained with two thermal analysis systems, models 2960 and Q10 both from TA Instruments, respectively. The purge gas was an air flow of 100 mL min^{-1} (TG-DTA) and 50 mL min^{-1} (DSC), a heating rate of 20 K min^{-1} for samples weighing about 7 mg (TG-DTA) and about 5 mg (DSC). Alumina and aluminium crucibles, the latter with perforated cover, were used for TG-DTA and DSC, respectively.

Carbon and hydrogen contents were determined by microanalytical procedures, with an EA 1110 CHNS-O Elemental Analyser from CE Instruments.

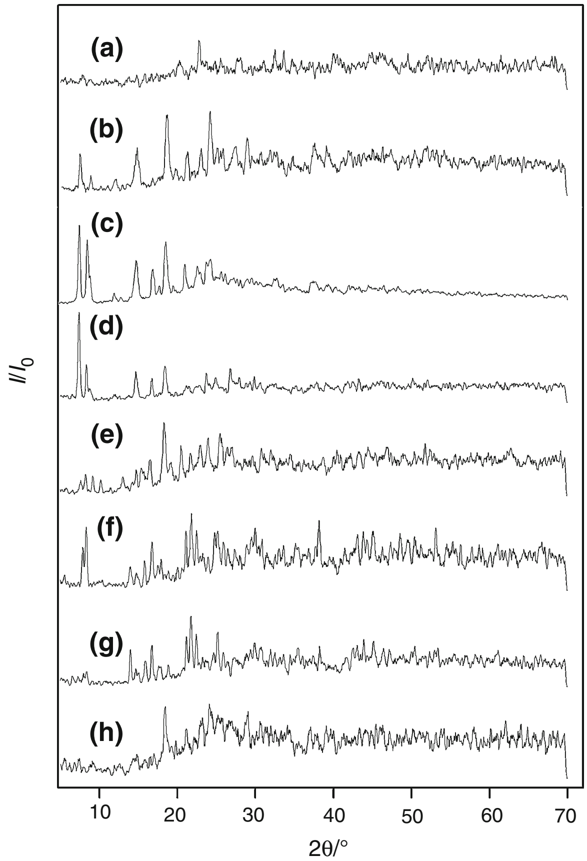

Fig. 1 X-ray powder diffraction patterns of the compounds: (*a*) $Tb(L)_3{\cdot}2H_2O$; (*b*) $Dy(L)_3$; (*c*) $Ho(L)_3{\cdot}0.5H_2O$; (*d*) $Er(L)_3{\cdot}1.5H_2O$; (*e*) $Tm(L)_3$; (*f*) $Yb(L)_3$; (*g*) $Lu(L)_3$; (*h*) $Y(L)_3$. L = 3-methoxybenzoate

Table 1 Analytical data for $Ln(L)_3{\cdot}nH_2O$

Compound	Water/%		Ligand lost/%		Metal oxide/%			Carbon/%		Hydrogen/%		Residue
	Calcd.	TG	Calcd.	TG	Calcd.	TG	EDTA	Calcd.	E.A.	Calcd.	E.A.	
$Tb(L)_3{\cdot}2H_2O$	5.56	5.34	65.61	65.76	28.83	28.90	28.45	44.45	44.55	3.89	3.86	Tb_4O_7
$Dy(L)_3$	–	–	69.72	69.46	30.28	30.54	30.09	46.80	46.63	3.44	3.43	Dy_2O_3
$Ho(L)_3{\cdot}0.5H_2O$	1.44	1.80	68.45	68.34	30.11	29.86	30.76	45.94	45.87	3.54	3.55	Ho_2O_3
$Er(L)_3{\cdot}1.5H_2O$	4.17	4.90	66.30	65.34	29.53	29.75	29.16	44.50	43.86	3.74	3.74	Er_2O_3
$Tm(L)_3$	–	–	69.00	68.91	31.00	31.09	30.33	46.31	46.25	3.41	3.41	Tm_2O_3
$Yb(L)_3$	–	–	68.55	68.09	31.45	31.91	31.17	46.00	45.69	3.39	3.37	Yb_2O_3
$Lu(L)_3$	–	–	68.34	68.35	31.66	31.65	32.08	45.87	45.88	3.38	3.38	Lu_2O_3
$Y(L)_3$	–	–	79.18	78.84	20.82	21.16	21.14	53.15	52.92	3.91	3.89	Y_2O_3

Ln, Tb(III); Dy(III); Ho(III); Er(III); Tm(III); Yb(III); Lu(III); Y(III); L, 3-methoxybenzoate

Table 2 Spectroscopic data for sodium 3-methoxybenzoate and compounds with lighter trivalent lanthanides

Compound	$\nu_{(O-H)}$ H_2O/ cm^{-1}	$\nu_{as(COO^-)}$/ cm^{-1}	$\nu_{s(COO^-)}$/ cm^{-1}	$\Delta\nu_{(\nu_{as}-\nu_s)}$/ cm^{-1}
Na(L)	–	1568_s	1400_s	168
$Tb(L)_3{\cdot}2H_2O$	3497_{br}	1537_s	1408_s	129
$Dy(L)_3$	3454_{br}	1533_s	1408_s	125
$Ho(L)_3{\cdot}0.5H_2O$	3452_{br}	1537_s	1406_s	131
$Er(L)_3{\cdot}1.5H_2O$	3454_{br}	1537_s	1408_s	129
$Tm(L)_3$	3439_{br}	1532_s	1410_s	122
$Yb(L)_3$	3435_{br}	1529_s	1412_s	117
$Lu(L)_3$	3429_{br}	1531_s	1414_s	117
$Y(L)_3$	3439_{br}	1535_s	1410_s	125

Br, Broad; s, strong; L, 3-metoxybenzoate; $\nu_{(O-H)}$, hydroxyl group stretching frequency; $\nu_{s(COO^-)}$ and $\nu_{as(COO^-)}$, symmetrical and anti-symmetrical vibrations of the COO^- structure

Results and discussion

The analytical and thermoanalytical (TG) data are shown in Table 1. These results establish the stoichiometry of these compounds, which are in agreement with the general formula $Ln(L)_3{\cdot}nH_2O$, where Ln represents trivalent Tb to Lu or Y, L is 3-methoxybenzoate and $n = 2$ (Tb), 1.5 (Er), 0.5 (Ho), 0 (Dy, Tm, Yb, Lu and Y).

The X-ray diffraction powder patterns (Fig. 1) show that all the compounds have a crystalline structure, without evidence for the formation of isomorphous compounds.

Infrared spectroscopic data on sodium-3-methoxybenzoate (Na-3-MeO-Bz) and its compounds with trivalent metal ions considered in this study are shown in Table 2. The investigation was focused mainly within the 1700–1400 cm^{-1} range because the region is potentially most informative in attempting to assign coordination

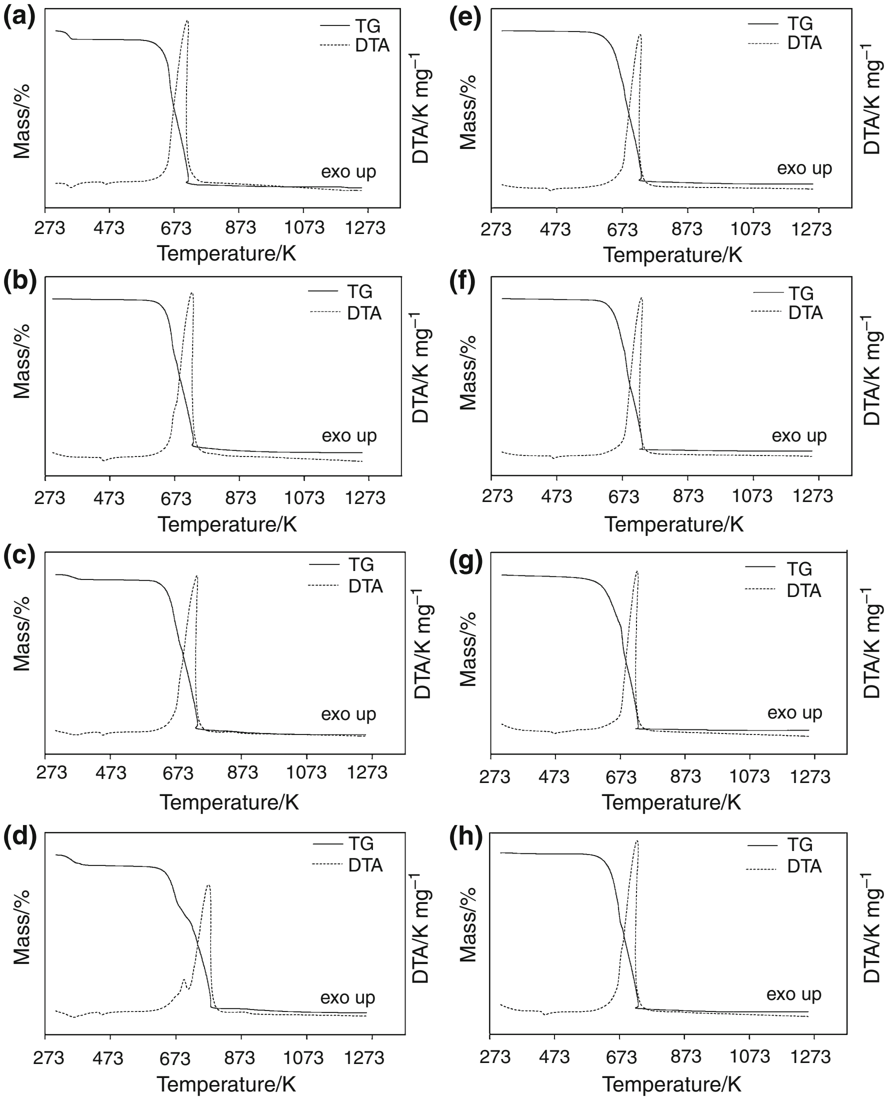

Fig. 2 TG-DTA curves of **a** $Tb(L)_3{\cdot}2H_2O$ ($m = 7.2435$ mg); **b** $Dy(L)_3$ ($m = 7.2150$ mg); **c** $Ho(L)_3{\cdot}0.5H_2O$ ($m = 7.4687$ mg); **d** $Er(L)_3{\cdot}1.5H_2O$ ($m = 7.3994$ mg); **e** $Tm(L)_3$ ($m = 7.1588$ mg); **f** $Yb(L)_3$ ($m = 6.9973$ mg); **g** $Lu(L)_3$ ($m = 6.9156$ mg); **h** $Y(L)_3$ ($m = 7.1948$ mg). L = 3-methoxybenzoate

sites. In the Na-3-MeO-Bz, strong bands located at 1568 and 1400 cm^{-1} are attributed to anti-symmetrical (ν_{as}) and symmetrical (ν_s) frequencies of the carboxylate groups, respectively [25, 26]. For the synthesized compounds, the anti-symmetrical and symmetrical stretching frequencies are located between 1537–1529 and 1414–1406 cm^{-1}, respectively. Analysis of the $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ bands shows that the lanthanides are linked to the carboxylate group by a bidentate bond with an incomplete equalization of bond lengths in the carboxylate anion, which is in agreement with [21].

Three patterns of thermal behaviour are observed for the anhydrous compounds. First, a close similarity is noted concerning the TG-DTA profiles of the dysprosium, holmium and yttrium compounds (Fig. 2b, c, h). The similarity is also observed for the terbium, thulium, ytterbium and lutetium compounds (Fig. 2a, e–g). On the other hand, erbium compound displays TG-DTA profiles without no similarity with the others, Fig. 2d.

Thus, the features of each of these compounds are discussed on the base of their similar thermal profiles of the anhydrous compounds.

Dysprosium, holmium and yttrium compounds

The simultaneous TG and DTA curves of these compounds are shown in Fig. 2b, c and h. These curves show mass loss in two (Dy, Y) or three (Ho) steps and thermal events corresponding to these losses or due to physical phenomenon. For the holmium compound (Fig. 2c), the first mass loss between 313 and 383 K, corresponding to the endothermic peak at 363 K, is due to dehydration with loss of 0.5 H_2O (Calcd. = 1.44%, TG = 1.80%).

The endothermic peak at 454 K (Dy), 450 K (Ho) and 442 K (Y), without mass loss in the TG curve, is attributed to the reversible crystalline phase transition, as can be seen in the DSC curve of terbium compound, Fig. 3, as representative of all compounds. The reversible crystalline phase transition had already been observed in the other lanthanide compounds [12–14].

Table 3 Temperature ranges/K, mass losses/% and peak temperature/K observed for each step of the TG-DTA curves of the $Ln(L)_3{\cdot}nH_2O$ compounds, where Ln = lanthanides(III) and yttrium (III), L = 3-methoxybenzoate

Compound	Dehydration	Steps (anhydrous compound)		
		First	Second	Third
$Tb(L)_3{\cdot}2H_2O$				
K	313–383	573–708	–	–
Loss/%	5.34	65.76	–	–
Peak/K	358	708	–	–
$Dy(L)_3$				
K	–	588–723	723–943	–
Loss/%	–	67.32	2.14	–
Peak/K	–	723	–	–
$Ho(L)_3{\cdot}0.5H_2O$				
K	313–383	598–738	738–973	–
Loss/%	1.80	65.67	2.67	–
Peak/K	363	738	848	–
$Er(L)_3{\cdot}1.5H_2O$				
K	318–393	598–708	708–773	773–993
Loss/%	4.90	23.41	40.29	1.64
Peak/K	364	698	773	908
$Tm(L)_3$				
K	–	563–728	–	–
Loss/%	–	68.91	–	–
Peak/K	–	728	–	–
$Yb(L)_3$				
K	–	593–733	–	–
Loss/%	–	68.09	–	–
Peak/K	–	733	–	–
$Lu(L)_3$				
K	–	548–723	–	–
Loss/%	–	68.35	–	–
Peak/K	–	723	–	–
$Y(L)_3$				
K	–	570–723	723–903	–
Loss/%	–	77.70	1.14	–
Peak/K	–	723	–	–

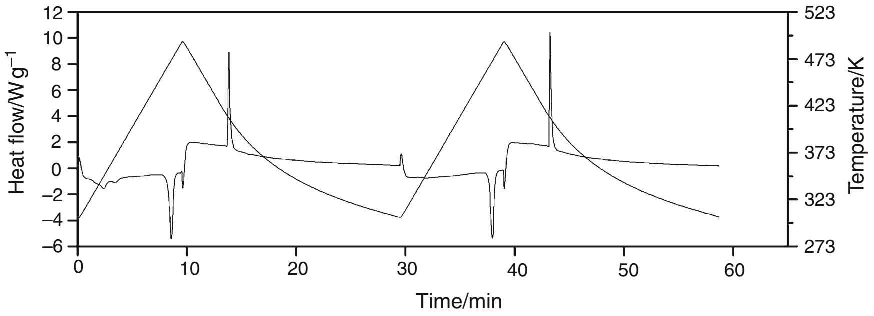

Fig. 3 DSC curves of heating and cooling of the compound $Tb(L)_3$ (m = 2.488 mg); purge gas: air; flow of 50 mL min^{-1}; heating rate of 20 K min^{-1}. L = 3-methoxybenzoate

The anhydrous compounds are stable up to 588 K (Dy), 598 K (Ho) and 570 K (Y), and above this temperature the thermal decomposition occurs in two steps. The first mass loss corresponding to a large exothermic peak is attributed to the oxidation of the organic matter with the formation of small amount of carbonaceous residue. The profiles of the TG-DTA curves in this step show that the oxidation of the organic matter occurs with combustion. In the second mass loss, only the holmium compound exhibit a small exothermic event, while no thermal event is observed in the DTA curve of the dysprosium and yttrium compounds; probably because the oxidation of the carbonaceous residue occurs slowly and the heat evolved in this step is insufficient to produce the thermal event.

Terbium, thulium, ytterbium and lutetium compounds

The simultaneous TG and DTA curves of these compounds are shown in Fig. 2a and e–g. The curves show mass loss in a single step (Tm, Yb and Lu) or two steps (Tb) and thermal events corresponding to these losses or due to physical phenomenon.

For the terbium compound, Fig. 2a, the first mass loss between 313 and 383 K corresponding to an endothermic peak at 358 K is due to dehydration with loss of 2 H_2O (Calcd. = 5.56%, TG = 5.34%). The endothermic peak at 458 K (Tb), 451 K (Tm), 461 K (Yb) and 465 K (Lu), without mass loss in the TG curve, is attributed to the reversible crystalline phase transition.

The anhydrous compounds are stable up to 573 K (Tb), 563 K (Tm), 593 K (Yb) and 548 K (Lu), and above this temperature the thermal decomposition occurs in a single step, corresponding to a large exothermic peak attributed to the oxidation of the organic matter. The profiles of the TG-DTA curves in this step also show that the oxidation of the organic matter occurs with combustion, with formation of the respective oxide, as final residue.

Erbium compound

The simultaneous TG and DTA curves are shown in Fig. 2d. These curves show mass loss in four steps. The first mass loss between 318 and 393 K corresponding to an endothermic peak at 364 K is due to dehydration with loss of 1.5 H_2O (Calcd. = 4.17%, TG = 4.90%). The endothermic peak at 446 K, without mass loss in the TG curve, is attributed to the reversible crystalline phase transition.

The anhydrous compound is stable up to 598 K, and above this temperature the thermal decomposition occurs in three steps, being the first two overlapping ones,

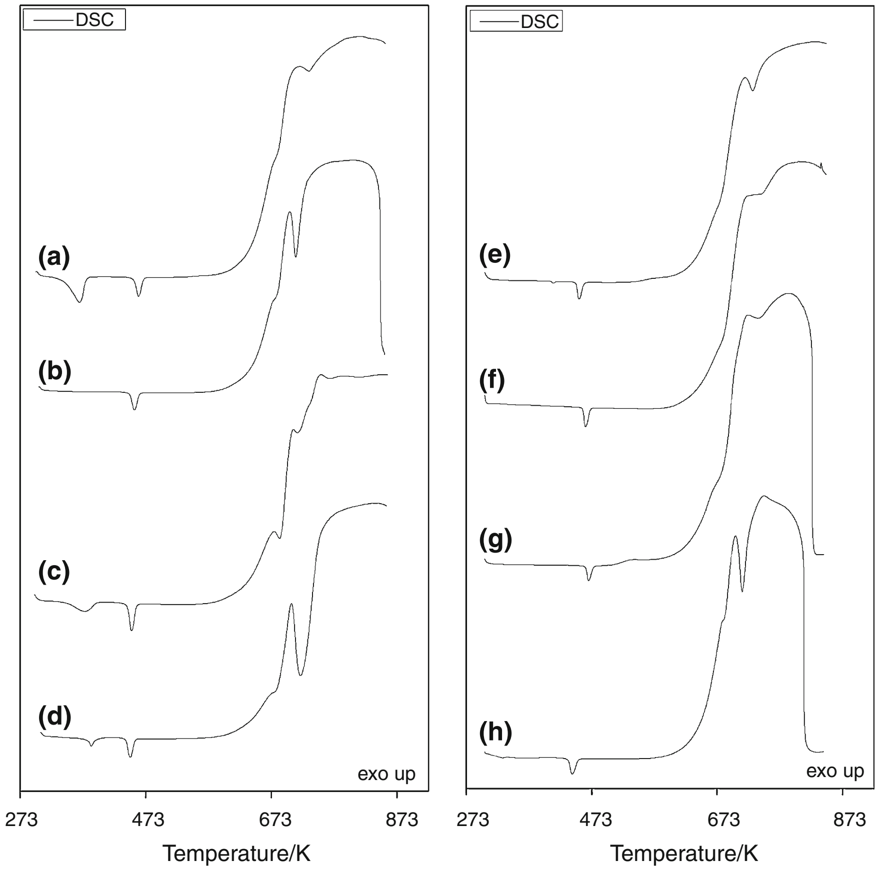

Fig. 4 DSC curves of: (*a*) $Tb(L)_3{\cdot}2H_2O$; (*b*) $Dy(L)_3$; (*c*) $Ho(L)_3{\cdot}0.5H_2O$; (*d*) $Er(L)_3{\cdot}1.5H_2O$; (*e*) $Tm(L)_3$; (*f*) $Yb(L)_3$; (*g*) $Lu(L)_3$; (*h*) $Y(L)_3$. L = 3-methoxybenzoate

corresponding to exothermic peaks attributed to the oxidation of the organic matter (first two) and oxidation of carbonaceous residue (last exothermic event), with formation of erbium oxide.

The mass loss (Δm), temperature range and peak temperature (K) observed in each step of TG and DTA curves, as well as the final residue of thermal decomposition of all the compounds, were collected in Table 3.

For all the compounds synthesized, the DSC curves are shown in Fig. 4. These curves show endothermic and exothermic events that all accord with the mass losses observed in the TG curves and endothermic peaks due to crystalline phase transition. The first endothermic peak observed at 368 K (Tb), 377 K (Ho) and 386 K (Er) is attributed to dehydration and in agreement with the TG-DTA curves. The endothermic peak at 461 K (Tb), 454 K (Dy), 450 K (Ho), 449 K (Er), 453 K (Tm), 463 K (Yb), 468 K (Lu) and 442 K (Y) is attributed to the reversible crystalline phase transition, and the other endothermic peak at 412 K observed only in the thulium compound is attributed to the irreversible crystalline phase transition, which was confirmed by X-ray powder diffraction, Fig. 5. The physical phenomena had already been observed in the other lanthanide compounds [12–14].

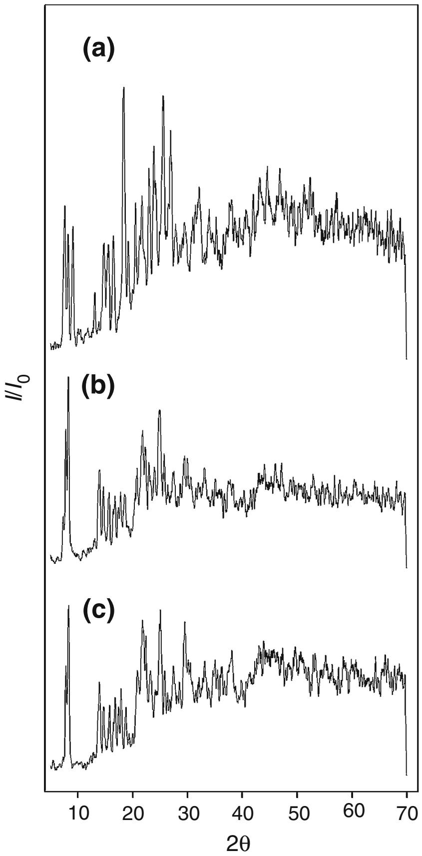

Fig. 5 X-ray powder diffraction patterns of the thulium compound heating up to: (*a*) 373 K; (*b*) 423 K; (*c*) 498 K

Table 4 Enthalpies in kJ mol^{-1} found for dehydration (A), reversible crystalline phase transition (B) and irreversible crystalline phase transition (C)

Compound	A	B	C
$Tb(L)_3 \cdot 2H_2O$	74.6	31.4	–
$Dy(L)_3$	–	30.7	–
$Ho(L)_3 \cdot 0.5H_2O$	26.5	33.7	–
$Er(L)_3 \cdot 1.5H_2O$	8.40	31.3	1.02
$Tm(L)_3$	–	19.6	–
$Yb(L)_3$	–	24.3	–
$Lu(L)_3$	–	20.9	–
$Y(L)_3$	–	32.6	–

L, 3-methoxybenzoate

The exothermic events are attributed to the thermal decomposition of the anhydrous compounds where the oxidation of the organic matter takes place in consecutive and/or overlapping steps.

The enthalpies belonging to dehydration, reversible crystalline phase transition and irreversible crystalline phase transition are shown in Table 4.

Conclusions

From TG curves and complexometry results, a general formula could be established for these compounds in the solid state.

The X-ray powder patterns pointed out that the synthesized compounds have a crystalline structure without evidence concerning to the formation of isomorphous series.

The infrared spectroscopic data suggest that the 3-methoxybenzoate acts as a bidentate ligand with an incomplete equalization of bond lengths in the carboxylate anion.

The TG-DTA and DSC curves provided previously underported information concerning the thermal behaviour and thermal decomposition of these compounds.

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