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Thermal decomposition of calcium(II) bis(acetylacetonate) n-hydrate

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Summary

The objective of this study was to investigate the thermal decomposition path of calcium bis(acetylacetonate) n-hydrate ($\text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2 \cdot n\text{H}_2\text{O}$). Thermal analysis of the investigated complex was accomplished using Simultaneous Differential Thermal and Thermo-Gravimetric Analysis (DTA/TGA) performed in dynamic air atmosphere up to 1,300°C. The obtained curves have been interpreted on the basis of ^1H and ^{13}C Nuclear Magnetic Resonance (NMR), Fourier-transformed Infra-Red (FTIR) Spectroscopy and X-Ray Diffraction (XRD) analysis of samples whose thermal treatment was interrupted at various temperatures. A total of six thermal decomposition steps, resulting in a total mass loss of 80%, has been observed: the release of adsorbed and coordinated water molecules in temperature intervals of 46-105°C and 105-175°C, respectively; two-step chelate decomposition processes in temperature interval of 175-305°C and 305-635°C, accompanied by the formation of calcite; and calcite decarboxylation and formation of calcium oxide in temperature interval between 635-800°C and oxidation of amorphous carbon residues at temperatures above 800°C.

KEYWORDS:

calcium bis(acetylacetonate)

FTIR spectroscopy

thermal decomposition

X-ray diffraction

KLJUČNE RIJEČI:

FT-IR spektroskopija

kalcijev bis(acetilacetonat)

rendgenska difrakcija

toplinski raspad

Termička razgradnja kalcijeva(II) bis(acetilacetonata) n-hidrata

Sažetak

Cilj ovog istraživanja bio je ispitati tijek termičke razgradnje kalcijeva acetilacetonata n-hidrata ($\text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2 \cdot n\text{H}_2\text{O}$). Toplinska analiza istraživanoga kompleksa provedena je simultanom diferencijalnom termičkom i termogravimetrijskom (DTA/TGA) analizom u struji zraka do 1 300 °C. Dobivene krivulje interpretirane su na temelju ^1H i ^{13}C nuklearne magnetske rezonancije (NMR), Fourierove pretvorne infracrvene spektrometrije (FT-IR) i rendgenske difrakcije (XRD) uzoraka čija je termička obrada prekidana pri različitim temperaturama. Ukupno je opaženo šest stupnjeva toplinskog raspada, koji su doveli do ukupnoga gubitka mase od 80 %: otpuštanje adsorbirane i kristalne vode

u temperaturnom intervalu od 46 do 105 °C i od 105 do 175 °C, dva procesa raspada kelata u temperaturnom intervalu od 175 do 305 °C i od 305 do 635 °C, praćena stvaranjem kalcita, te dekarboksilacija kalcita i formiranja kalcijeva oksida u temperaturnom intervalu između 635 i 800 °C i oksidacija zaostalog amorfog ugljika pri temperaturama iznad 800 °C.

Introduction

Metal acetylacetonates are coordination complexes with typical formula $\text{M}^{n+}(\text{AcAc})_n$ where AcAc is abbreviation for acetylacetonate anion, ($\text{C}_5\text{H}_7\text{O}_2$). Most often acetylacetonate acts as bidentate ligand, i.e. both oxygen atoms of acetylacetonate anion bind to the metal to form a six-membered chelate ring.¹ A great number of metal acetylacetonates have been prepared up to now.² These compounds are characterized by thermal stability, volatility and solubility in organic solvents.³ Such properties of metal acetylacetonates have opened possibilities for many uses, particularly in the area of nanotechnology.^{4,5,6} Metal acetylacetonates, together with other β -diketonate and β -ketoester chelate compounds are common precursors for the preparation of pure metals or oxides nanoparticles,⁴ chemical vapor deposition (CVD) of metal and non-metal thin films⁵ and sol-gel synthesis of powders and thin films.⁶ In addition to the mentioned applications calcium acetylacetonate is the most ordinary heat stabilizer for halogenated polymers such as PVC.⁷ It can also be used as catalyst, cross-linking agent, resin hardening accelerant, resin and rubber additive, etc.⁷

The information on thermal stability, thermal decomposition process and decomposition products is important for a lot of mentioned applications. Therefore, the purpose of this work is to investigate the thermal behaviour of Ca acetylacetonate. A better understanding of the thermal behaviour would enable better control of processes involving this compound.

Experimental part

Calcium bis(acetylacetonate) n-hydrate ($\text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2 \cdot n\text{H}_2\text{O}$) was prepared by dissolving 5 g (0.02 mol) of calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) and 7 g (0.007 mol) of 2,4-pentadione (acetylacetonate, $\text{C}_5\text{H}_8\text{O}_2$) in 25 ml of distilled water. Simultaneously, ammonium hydroxide was added drop-wise into the reaction solution. The pH was carefully controlled in order to reach and maintain pH = 9. The precipitation occurred, the precipitate was collected and washed three times with distilled water and dried. The synthesis was repeated once in order to check the reproducibility.

^1H and ^{13}C NMR spectra of obtained sample were recorded in high-resolution NMR Spectrometer Bruker Avance 300, operating at 300 MHz for ^1H resonance and at 75 MHz for ^{13}C resonance. The samples were dissolved in dimethyl sulfoxide (DMSO) containing tetramethylsilane as the internal standard and measured in 5 mm NMR tubes. The ^1H and ^{13}C NMR chemical shift values (δ) are expressed in ppm referring to tetramethylsilane. Assignments of ^1H and ^{13}C NMR spectra were performed on the basis of chemical shifts, signal intensities and literature references. The quantitative data were obtained by fitting the resonances to the Gauss function.

The thermal evolution of gel was characterized with DTA-TGA thermal analysis (Netsch STA 409C thermo analyzer). For the thermal analysis

~50 mg of material were placed in Pt crucibles and heated to 1,300 °C at a rate of 10 °Cmin⁻¹ in a synthetic air flow of 30 cm³min⁻¹. α -alumina was used as reference.

The IR spectra of the samples were acquired using the Fourier transform infrared spectrometer *Bruker Vertex 70* in ATR (attenuated total reflectance) mode. The samples were deposited on a diamond and the absorbance data were collected between 400 and 4,000 cm⁻¹ with a spectral resolution of 1 cm⁻¹ and an average of 64 scans.

The powder crystal X-ray diffraction (XRD) was accomplished using *Shimadzu diffractometer XRD 6000* with CuK α radiation. Data were collected between 5 and 65 °2 θ in a step scan mode with steps of 0.02° and counting time of 0.6 s. All analyses except NMR were done at least twice yielding no notable difference.

Results and Discussion

The identity and purity of the prepared compound was confirmed using NMR. ¹H and ¹³C NMR spectra of prepared sample are presented in Figure 1. Acetylacetone is capable of undergoing keto-enol isomerization, existing at room temperature as a mixture of the two tautomers (Figure 2a). The hydroxylic proton is easily ionized (Figure 2b) and the negative charge of the resulting anion is delocalized over the molecule (Figure 2c). The formed anion acts as bidentate ligand which chelates to the metal forming six-membered ring⁸ (Figure 2d).

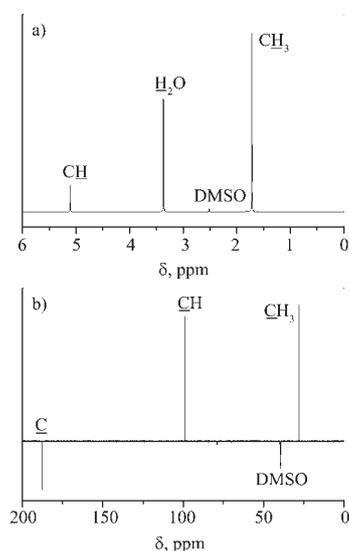


FIGURE 1 – a - ¹H and b - ¹³C NMR spectra of the prepared chelate in DMSO

Therefore, the ¹H NMR spectrum of sample exhibits signals due to an enolic form of acetylacetone: singlets of methyne proton at 5.11 and methyl protons at 1.71 ppm, clearly revealing the occurrence of chelation. At the same time, judging by the absence of characteristic keto tautomer signals, NMR spectroscopy confirmed that no uncoordinated free ligand was present. Additionally, the water proton resonance appears at 3.37 ppm. The fitting of ¹H NMR resonances to Gauss function yielded the quantitative relationship of Ca(AcAc)₂ to H₂O of approximately 1:3.3.

The ¹³C NMR spectra of the investigated samples showed the expected number of signals, which are in good correlation with the ¹H NMR spectra. The signals due to an enolic form of AcAc bonded to Ca were as follows: methyl carbons at 27 ppm, methyne carbon at 99 ppm and quaternary carbons at 188 ppm. The ¹³C NMR spectroscopy also confirmed that acetylacetone was present solely as the enolic tautomer coordinated to calcium.

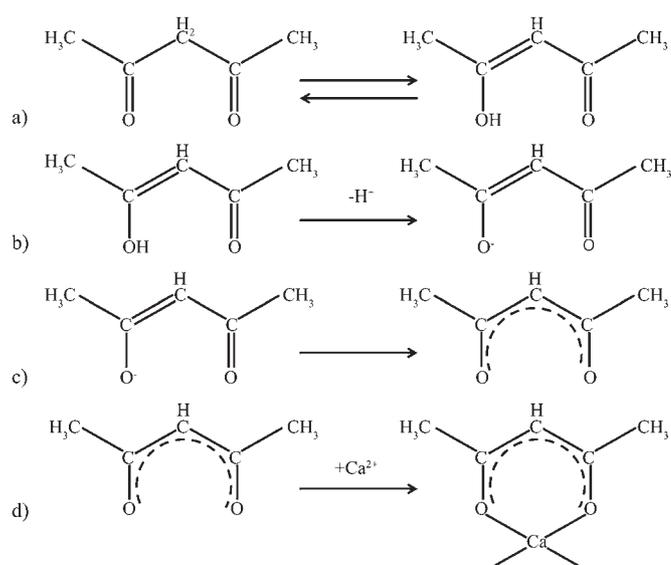


FIGURE 2 – a - keto-enol tautomerism of acetylacetone; b - formation of acetylacetonate anion; c - delocalization of negative charge, d - formation of the six-membered chelate ring

In order to gain insight into thermal stability of the precursor, a simultaneous differential thermal and thermo-gravimetric analysis has been conducted. Figure 3 displays the DTA, TGA and DTG curves of calcium acetylacetonate n-hydrate in dynamic air atmosphere up to 1,300°C. Taking into consideration all three curves, six processes could be observed. The first process is characterized by endothermic curve centered at 82°C accompanied with the mass loss of 15%. The second process is characterized by two overlapping endothermic curves centered at 119 and 142°C accompanied with mass loss of 3.2%. The third process yields a combination of endothermic and exothermic peak centered at 285°C, which is clearly visible from the inset in Fig 3. A similar combination, but associated with slower process could be observed centered at 368°C. The third and fourth process are accompanied by mass loss of 21.2 and 19.6%, respectively. The fifth process could be observed as wide endothermic peak centered at 764°C and associated with mass loss of 17.7%. Finally, in the temperature interval between ~800 and 1,300°C a continuous mass loss of 3.3% could be observed.

In order to characterize the intermediates and final products of the chelate sample thermal decomposition and to attribute the observed processes, samples were quenched from various temperatures in a range of 100 to 800°C and analyzed using FTIR and XRD.

Figure 4 shows the FTIR spectra of the as-prepared sample and samples thermally treated at various temperatures. The spectrum of as-prepared sample is characterized by the bands due to acetylacetonate and water. The bandwidth centered at 3,183 cm⁻¹ is due to the asymmetric and symmetric O–H stretching vibrations⁹ pointing out to the presence of water. The absorption frequencies between 2,989 and 2,961 cm⁻¹ are the result of C–H stretching due to asymmetrical (2,989 cm⁻¹, 2,958 cm⁻¹) and symmetrical (2,916 cm⁻¹) stretching vibrations of the methyl group.^{10,11,12} While C = O stretching vibrations of keto form appear at wavenumbers above 1,700 cm⁻¹, those of enol form stabilized by chelation to metal atoms are shifted below 1,600 cm⁻¹.¹³ Therefore, the formation of chelate results in the appearance of absorption bands at 1,596 and 1,510 cm⁻¹ due to conjugated C=O vibrations in a complex ring.^{11,12} The spectrum also exhibits bands at 1,460 cm⁻¹ due to the asymmetrical deformation vibration of CH₃ groups^{10,11,2} and at 1,385 cm⁻¹ due to the symmetrical deformation vibration of CH₃ groups.^{10,11} The band at 1,360 cm⁻¹ is attributed to C=C stretching^{10,12} while the one at 1,243 cm⁻¹ is attributed to C=C=C stretching in complex ring.¹² The band at 1,197 cm⁻¹ is due

in plane C-H bending mode^{10,11} while the band at $1,013\text{ cm}^{-1}$ is due to CH_3 rocking.^{10,12} The band at 914 cm^{-1} is due to C- CH_3 stretching^{10,11,12} and band at 764 cm^{-1} is due to out-of-plane C-H bending mode.^{10,12} The band at 655 cm^{-1} is due to ring deformation modes.^{10,11,12} FTIR spectra of as-prepared sample (Figure 4) is consistent with the structure having bifunctionally coordinated acetylacetonate anions and is in complete concordance with NMR spectra.

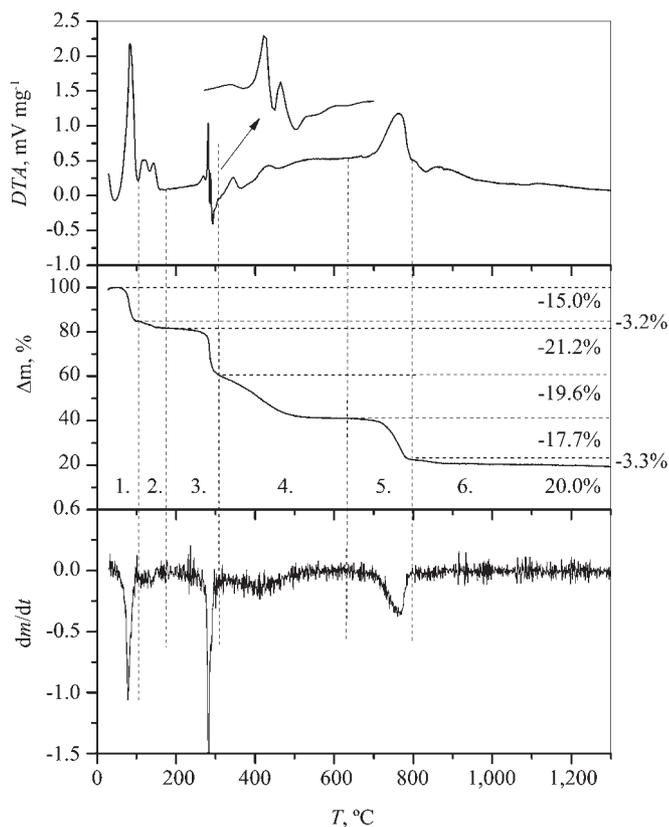


FIGURE 3 – DTA, TGA and DTG curves of the prepared chelate sample obtained with heating rate of 10°Cmin^{-1} in dynamic air

The only difference of FTIR spectra thermally treated at 100 and 200°C , compared to the as-prepared sample, is the weakening in intensity of a broad absorption band in the region of $3,500\text{--}2,500\text{ cm}^{-1}$, characteristic to water molecules. In those spectra there are no changes in the position and intensity of chelate absorption bands. Therefore, the first two endothermic processes and accompanied mass losses of 15 and 3.2% could be attributed to the evolution of water: the first of them, below 100°C , to the removal of adsorbed water and the second one, above 100°C , to the expulsion of coordinated water. The spectra of sample thermally treated at 300°C showed weakened intensity of chelate bands, indicating reduced content of calcium bis(acetylacetonate). The weakening of chelate bands intensity continues in samples thermally treated at 400 and 500°C and bands vanish completely in sample thermally treated at 600°C . Therefore, the third and fourth processes and mass losses of 21.2 and 19.6%, associated with them, could be ascribed to the decomposition of calcium bis(acetylacetonate) and burning of produced volatiles. On the other hand, bands characteristic for calcium carbonate ($1,460$; $1,400$; 871 and 713 cm^{-1}),^{14,15} a product of calcium acetylacetonate decomposition process, appears on the spectrum of the sample thermally treated even as low as 300°C . Bands grow in intensity with the increase of samples thermal treatment temperature, being the most intense for the sample thermally treated at 600°C , and diminish almost completely in the spectrum of sample thermally treated at 800°C . According to the literature, bands due to calcium oxide appear at $2,513$ and $1,796\text{ cm}^{-1}$,¹⁶ but those bands

were not observed. Although bands typical for calcium oxide were not observed, it is obvious that carbonate decomposes to calcium oxide and carbon dioxide yielding broad endotherm and mass loss of 17.7%. The sample thermally treated at 800°C is grayish which indicates the presence of carbon residue. Therefore, the last process, accompanied by the mass loss of 3.4%, obviously corresponds to the combustion of elemental carbon.

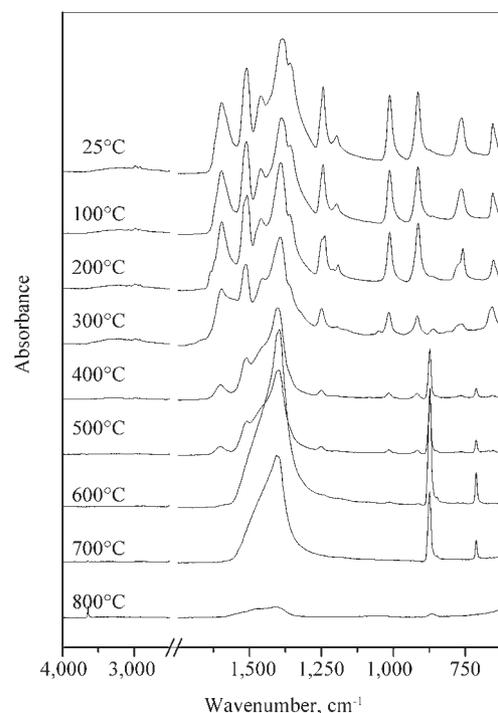


FIGURE 4 – FTIR spectra of the as-prepared sample and samples quenched from various temperatures

Powder XRD pattern of the as-prepared sample (Figure 5) is in concordance with the pattern of calcium bis(acetylacetonate) of unknown crystal system reported by Engwoll.¹⁷ The agreement could be characterized as fairly good, as far as peak angles are concerned. On the other hand, there is no agreement regarding intensities. Moreover, a drastic change in relative intensities of individual peaks in the XRD patterns of samples quenched from 200°C , in comparison with as-prepared sample and sample quenched from 100°C , could be observed. Most often the anomalous changes in relative intensities of individual XRD peaks are the consequence of the preferred orientation. On the other hand, drastic changes in intensity of individual peaks in the XRD patterns can result from changes in distribution of electron density within the unit cell. The electron density distribution could depend on the presence and nature of guest molecules, such as H_2O molecule.¹⁸ Therefore, the changes in XRD patterns of samples quenched from 200°C in comparison with the pattern of as-prepared sample and sample quenched from 100°C are interpreted as a consequence of removal of water molecules from the structure.

Sample thermally treated at 300°C is almost completely amorphous; on closer inspection only very weak peaks due to calcite could be observed. Therefore, XRD points out complete decomposition of calcium bis(acetylacetonate) crystalline phase. It is interesting to note that in FTIR spectra of the same sample show rather intense chelate bands since, although crystal structure has decomposed, a lot of acetylacetonate is still bonded to calcium.

With the increase of the thermal treatment temperature the intensity of carbonate peaks grow, being the most intense for the sample thermally

TABLE 1 – The assignments of IR bands in Figure 4

Wavenumber, cm ⁻¹	Assignment	Wavenumber, cm ⁻¹	Assignment
Ca(AcAc) ₂		H ₂ O	
2,989	$\nu(\text{CH}_3)_{\text{as}}$	3,183	$\nu(\text{O-H})$
2,958	$\nu(\text{CH}_3)_{\text{as}}$		
2,916	$\nu(\text{CH}_3)_{\text{s}}$	CaCO ₃	
1,596	$\nu(\text{C}=\text{O})$	1,460	$\nu(\text{CO}_3)_{\text{as}}$
1,510	$\nu(\text{C}=\text{O})$	1,400	$\nu(\text{CO}_3)_{\text{as}}$
1,460	$\delta(\text{CH}_3)_{\text{as}}$	871	$\nu(\text{CO}_3)_{\text{as}}$
1,385	$\delta(\text{CH}_3)_{\text{s}}$	713	$\nu(\text{CO}_3)_{\text{s}}$
1,360	$\nu(\text{C}=\text{C})$		
1,243	$\nu(\text{C}=\text{C}=\text{C})$	CaO	
1,197	$\beta(\text{C-H})$	2,513	$\nu(\text{CaO})$
1,013	$\rho(\text{CH}_3)$	1,796	$\nu(\text{CaO})$
914	$\nu(\text{C-CH}_3)$		
764	$\gamma(\text{C-H})$		
655	$\delta(\text{ring})$		

ν - stretching mode, β - in plane bending mode, γ - out-of plane bending mode, ρ - rocking mode, δ - deformation mode

treated at 600°C and diminish almost completely in the pattern of the sample thermally treated at 800°C. At the same time bands due to calcium oxide appear. These observations are in complete concordance with the conclusions drawn from FTIR spectra interpretation.

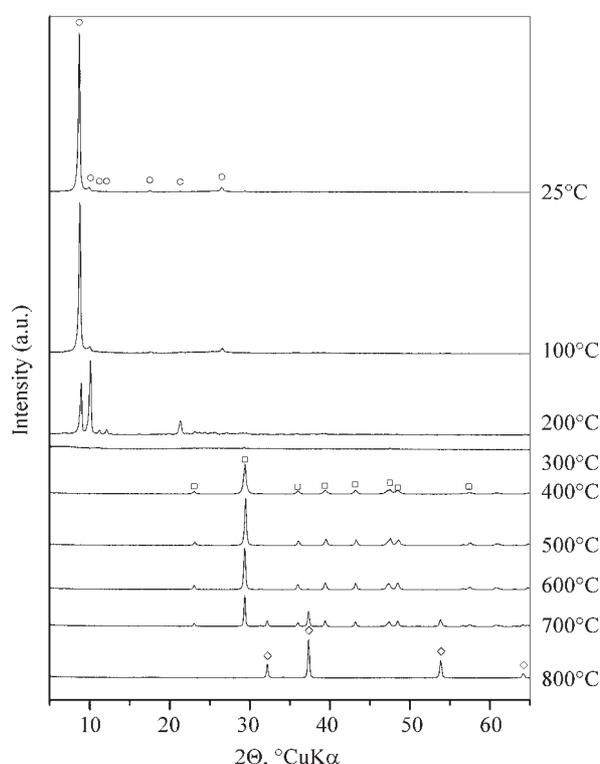


FIGURE 5 – Powder XRD pattern of the as-prepared sample and samples quenched from various temperatures. ○ - Calcium bis(acetylacetonate) n-hydrate¹⁷, □ - calcite (ICDD-PDF No. 72-1214), ◇ - calcium oxide (ICDD-PDF No. 77-2376)

On the basis of the results of DTA/TGA, FTIR and XRD analysis, taking into consideration that no sublimation has been noted, reducing all pos-

sible acetylacetonate oxidation products (such as CO, acetone and acetic acid¹⁹) to carbon dioxide and water, and assuming that amorphous carbon residue is a consequence of a faster oxidation process, the thermal decomposition path of calcium bis(acetylacetonate) n-hydrate could be written as follows:

- $\text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2 \times 0.6\text{H}_2\text{O} + 2.2\text{H}_2\text{O} \xrightarrow{-2.2\text{H}_2\text{O}} \text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2 \times 0.6\text{H}_2\text{O}$
- $\text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2 \times 0.6\text{H}_2\text{O} \xrightarrow{-0.6\text{H}_2\text{O}} \text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2$
- $\text{Ca}(\text{C}_5\text{H}_7\text{O}_2)_2 \xrightarrow{+5.5\text{O}_2 - 4\text{CO}_2 - 3.5\text{H}_2\text{O}} \text{Ca}(\text{C}_5\text{H}_7\text{O}_2)(\text{CO}_3)_{0.5} + 0.5\text{C}$
- $\text{Ca}(\text{C}_5\text{H}_7\text{O}_2)(\text{CO}_3)_{0.5} + 0.5\text{C} \xrightarrow{+5.5\text{O}_2 - 4\text{CO}_2 - 3.5\text{H}_2\text{O}} \text{CaCO}_3 + \text{C}$
- $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$
- $\text{C} + \text{O}_2 \rightarrow \text{CO}_2$

Mass balance confirming the presented thermal decomposition path is given in Table 2.

As can be seen, normalization of final residue to 1 mol of calcium oxide points out to initial composition of approximately $\text{Ca}(\text{AcAc})_2 \times 2.8\text{H}_2\text{O}$. In the course of the first process, in the temperature range from room temperature to 105°C, the material undergoes endothermic dehydration losing approximately 2.3 mol of water. The second, also endothermic, process (i.e. two consecutive processes) is due to the expulsion of coordinated water; according to TGA in the course of this process the material losses are approximately 0.5 mol of water. $\text{Ca}(\text{AcAc})_2$ is coordinatively unsaturated and water can give the octahedral adduct, $\text{Ca}(\text{AcAc})_2 \times 2\text{H}_2\text{O}$. Since only 0.5 water molecule per one chelate molecule has been detected it is most probable that octahedral calcium coordination has been reached through oligomerization. Investigations of $\text{Ca}(\text{AcAc})_2$ and $\text{Ni}(\text{AcAc})_2$ provided evidence that those compounds associate to form oligomeric complexes in which some oxygen centres bridge two cations.^{20,21}

Mass loss in the course of the third process is approximately equivalent to the difference between one mole of AcAc and one mole of CO₂. Rather unusual shape of DTA peak centered at 285°C is the consequence of convolution of endothermal peak due to the decomposition process and exothermal peak due to combustion of released decomposition products. According to XRD pattern, the third process results in amorphous inter-

TABLE 2 – Mass balance of the prepared chelate, based on DTA and TGA curves, FTIR scans and XRD patterns, of chelate samples quenched from various temperatures

Process	Temp. Interval, °C	Centered at, °C	Δm , %	Species lost	M, gmol ⁻¹	n ^a , mol	n ^b , mol
1.	46-105	82	15	H ₂ O	18	0.83	2.31
2.	105-175	119, 142	3.2	H ₂ O	18	0.18	0.50
3.	175-305	285	21.2	4C+7H	55	0.38	1.06
4.	305-635	368	19.6	4C+7H	55	0.36	1
5.	635-800	764	17.7	CO ₂	44	0.40	1.11
6.	800-1,300		3.3	C	12	0.28	0.78
residue	1,300		20.0	CaO	56	0.36	1

^aassuming initial mass of 100 g; ^bnormalized to 1 mol of CaO

mediate product. The fourth process, centered at 368°C, is equivalent to the third, except that this process is rather slow, stretched roughly between 300 and 600°C. Instead of abrupt decomposition followed by the burning of volatiles, a slow decomposition and oxidation process occurs. An additional contribution to the overall energetic is given with calcite crystallization, so the fourth crystallization peak is convolution product of decomposition, oxidation and crystallization. It could be postulated that in the course of both processes, especially in the course of the later, rather sluggish process, some amorphous carbon accumulates due to incomplete oxidation. No significant sublimation process has been observed. Low volatility could be taken as additional indication of oligomerization since self-associated chelates have low volatility and are thermally dissociated when heated.²²

The residue left after complete chelate decomposition, on the basis of XRD pattern, is found to be pure calcite. Although analyses were not conducted in a quantitative manner, it could be argued that at 600°C both, FTIR spectra and XRD pattern, point out to maximal amount of calcite. Therefore, according to DTA/TGA, FTIR and XRD, the fifth process, calcite decomposition, begins roughly at 600°C and ends roughly at 800°C yielding broad endotherm and approximate loss of 1 mol of CO₂. From this temperature onward, traces of amorphous carbon residue are oxidized and expelled, yielding the mass loss equivalent roughly to 0.8 mol of carbon. Of course, it is most likely that carbon oxidation process is stretched between 300 and 1,300°C. While it can be quantitatively determined for the interval of 800°C onward, where there are no other processes, the extent of this process in 300 to 800°C temperature interval remains unknown. The final product is a white crystalline powder identified as CaO.

Conclusion

The thermal behavior of calcium bis(acetylacetonate) n-hydrate has been investigated. According to ¹H and ¹³C NMR, as well as simultaneous DTA and TGA analyses, the chelate comprises various amounts of adsorbed and approximately 0.6 mol of coordinated water molecules being removed in temperature intervals of 45-105°C and 105-175°C, respectively. Under dynamic air atmosphere calcium bis(acetylacetonate) is thermally stable up to 175°C, when it starts to decompose in two distinct steps centered at 285 and 386°C forming calcium carbonate, calcite. The calcium carbonate so formed undergoes decarboxylation in the temperature interval between 635-800°C and consequently forms calcium oxide. Traces of amorphous carbon accumulated due to incomplete oxidation in previous steps are oxidized up to 1,300°C. The total of six thermal decomposition steps, resulting in a total mass loss of 80%, have been observed.

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IZLOG KNJIGA

Friedrich Kurr Praxishandbuch der Qualitäts- und Schadensanalyse für Kunststoffe



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Sadržaj: *Vorworte und Hinweise zum Gebrauch; Fachwortverzeichnis von Qualitäts- und Schadenbegriffen; Definitionen der Fachworte aus Kapitel 1 und 3; Qualitäts- und Schadenbilder; Anhang*

Autor ove knjige Friedrich Kurr ima svestranu naobrazbu kao mehaničar, alatničar, strojar-ski tehničar i diplomirani inženjer na području

strojogradnje. Bio je suradnik u *Süddeutschen Kunststoff-Zentrum (SZK)* te predavao za stručnjake iz područja polimerstva u drugim ustanovama, između ostalog na *Sveučilištu u Erlangenu i Tehničkim akademijama* u Esslingenu i Sarnenu (Švicarska) te na *Visokoj školi* u Würzburgu. Mnogim posjetima industriji i sajmovima ostvario je izvanredne kontakte s plastičarskom i gumarskom industrijom. Osnovao je *Laboratorij za analizu kvalitete i oštećenja primjenom makroskopa, univerzalnog mikroskopa i raster-elektronskog mikroskopa*. Niz godina slovio je kao stručnjak na području umjetnog starenja te analize kvalitete i oštećenja polimera. Nakon odlaska u mirovinu sazeo je svoje bogato dugogodišnje iskustvo u ovu knjigu na traženje mnogih nekadašnjih korisnika njegovih ispitivanja. Knjiga sadržava više od 2 620 pojmova iz područja ispitivanja kvalitete i oštećenja polimera, kao i objašnjenja njihovih uzroka. Sve je to ilustrirano s 588 slika mikroskopskih analiza ispitnih tijela, poluproizvoda i gotovih dijelova. Slike su podijeljene u 74 područja prerade polimera i primjene uz uobičajene nazive, abecedno poredane.

Prvo poglavlje sadržava abecedno poredane stručne nazive iz mnogih područja polimerstva s uputama o tom pojmu, brojem slike i pripadajućim slikama vezanim uz kontrolu

kvalitete ili oštećenja. Brojevi nakon navedenoga stručnog pojma upućuju na tekst kojim se taj pojam objašnjava u poglavlju 2. Sve je to na neki način samo uvod u najvrjednije, treće poglavlje u kojem su s pomoću 588 slika dobivenih svjetlosnim ili raster-elektronskim mikroskopom objašnjeni uzroci oštećenja. Primjeri su iz različitih područja, kao npr. pojava napuklina na brtvi od etilen/propilen/dienskog kaučuka (EPDM) za prozorske profile od poli(vinil-klorida) (PVC) nakon umjetnog starenja u trajanju od 4 074 sata ili pak ostaci kirurškog konca kako bi se procijenila njihova razgradljivost u određenim vremenskim razdobljima. Ili ilustrativno prikazani primjeri problema zavarivanja polimera. U svakom pogledu izuzetno vrijedna podloga za sve koji se bave polimernim materijalima.

Prema riječima samog autora, knjiga nudi praktične primjere koji omogućuju dovoljno saznanja da se u najkraćem vremenu, samo uz makroskop i mikroskop, često bez skupih uređaja i uz prihvatljive troškove, mogu riješiti mnogi problemi. U tom obliku je ovakva knjiga, temeljena na praksi, novost u području osiguranja kvalitete i mnogi će, bez sumnje, u njoj naići na brojne slične primjere iz vlastite prakse.

Đurđica ŠPANIČEK