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Principal component analysis of thermal decomposition of magnesium salts used as drugs

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Abstract Thermal decomposition of magnesium salts of organic acids used in medicine (Mg acetate, Mg valproate, Mg lactate, Mg citrate, Mg hydrogen aspartate, Zn hydrogen aspartate) was analyzed by thermoanalytical, calorimetrical, and computational methods. Thermoanalytical studies were performed with aid of a derivatograph. 50-, 100-, and 200-mg samples were heated in a static air atmosphere at a heating rate of 3, 5, 10, and 15 °C min^{-1} up to the final temperature of 700–900 °C. By differential thermal analysis (DTA), thermogravimetry (TG), and derivative thermogravimetry (DTG) methods, it has been established that thermal decomposition of the salts under study occurs via two stages. The first stage (dehydratation) was distinctly marked on the thermoanalytical curves. Calorimetrical studies were carried out by using of a heat-flux Mettler Toledo differential scanning calorimetry (DSC) system. Ten milligram samples of compounds under study were heated in the temperature range from 20 to 400 °C at a heating rate of 10 and 20 °C min^{-1} under an air stream. The studies showed that the values of transitions heats and enthalpies of dehydration for investigated salts varied with the increasing of heating rate. For chemometric evaluation of thermoanalytical results, the principal component analysis (PCA) was applied. This method revealed that points on PC1 versus PC2 diagrams corresponding to the compounds of similar chemical constitution are localized in the similar ranges of the first two PC's values. This proves that thermal decomposition reflects similarity in the structure of magnesium salts of organic acids.

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Introduction

Pharmacologically active compounds are used in medicine more often in a solid drug form than in a form of a solution [1]. Because of the reason given above, their analysis in a solid form is an important issue. It is crucial both in relation to drug substances used in a process of drug formulation and also in the case of a substance stored for a long period of time, for instance, as a ready to use form of drug.

For investigation of stability and decomposition of organic drug substances, often the methods of thermal analysis are applied [2]. They enable recognition of the behavior of a given compound in different temperature conditions. It also makes possible to determine optimum conditions of storage of drugs and to define the parameters of technological processes, which can be used without loss of specific physicochemical properties of a drug [3, 4]. Moreover, the thermoanalytical methods assure conditions for conducting of a decomposition reaction to obtain solid products with a proper phase composition and activity, useful in further technological applications.

Taking all of this into consideration, the work's objective is to recognize the characteristics of decomposition of magnesium salts applied in medicine, using the obtained information for the stability evaluation of these substances in a solid phase, as well as to determine the influence of a chemical structure on the decomposition course. The assumed aim of the work was realized by determination of the course of thermal decomposition of the studied salts, analysis of influence of the heating rate, and of the sample

mass on the course of thermal degradation, as well as by studying of influence of a chemical structure of a substance on thermal decomposition course by the use of multidimensional technique, principal component analysis (PCA).

In the literature, there are few examples of the studies, in which magnesium salts of organic acids were investigated by thermoanalytical methods. Among organic acids, the thermal decomposition of magnesium salts of acetic [5, 6], malonic [7], stearic [8, 9], and salicylic [10] acids were studied by using differential scanning calorimetry (DSC), differential thermal analysis (DTA), and thermogravimetry (TG) methods. Moreover, the thermal properties of some complexes of magnesium with organic ligands, which are important form the biological point of view, e.g., 4-methoxybenzylidenepyruvate [11], nicotinamide [12], pyridine [13], ciprofloxacin [14], nitro-substituted benzoic acids [15], methyl-3-pyridyl carbamate [16] as well as alanine- and taurine-salicylal Schiff base [17], were also studied.

Experimental

Materials

The following magnesium salts (also zinc in the case of aminosuccinic acid), manufactured at various enterprises in Poland, were used in the studies (manufacturers are given in the parenthesis): magnesium acetate, magnesium methanecarboxylate, $Mg(CH_3COO)_2{\cdot}4H_2O$ (POCh, Gliwice); magnesium valproate, dipromal, magnesium 2-propylpentanate, $Mg[C_4H_8(C_3H_7)COO]_2{\cdot}H_2O$ (ICN POLFA, Rzeszow); magnesium lactate, magnesium α-hydroxypropionate $Mg[CH_3CH(OH)COO]_2{\cdot}H_2O$ (SANOFI-BIOCOM, Rzeszow); magnesium citrate, magnesium 2-hydroxy-1,2,3-propanetricarboxylate, $Mg_3[HOC(CH_2COO)_2COO]_2$ (KRKA, Warszawa); magnesium hydrogen aspartate, magnesium hydrogen aminosuccinate, magnesium hydrogen 2-aminoethylenedicarboxylate, $Mg[NH_2CH(COOH)CH_2COO]_2{\cdot}4H_2O$ (NOVI-CHEM, Chorzow); zinc hydrogen aspartate, zinc hydrogen aminosuccinate, zinc hydrogen 2-aminoethylenedicarboxylate, $Zn[NH_2CH(COOH)CH_2COO]_2$ (FARMAPOL, Poznan). All compounds were analyzed without further purification.

Methods

DTA, TG, and derivative thermogravimetry (DTG) curves of thermal decomposition of the studied compounds were recorded using the OD-103 derivatograph (MOM, Budapest, Hungary). 50, 100, and 200 mg samples were heated in an unsealed platinum crucible at a heating rate of 3, 5, 10, and 15 °C min^{-1} up to the final temperature of 700–900 °C. Each analysis in a static air atmosphere was repeated at least three times. α-Al_2O_3 was employed as reference material.

From DTA curves, the temperatures of onset (T_o), end (T_e), peak (T_p), and range (ΔT) of endo- and exothermic peaks in the successive stages of thermal decomposition of the studied compounds were acquired. In the case of TG curves, the temperatures of onset (T_o) and end (T_e) of the successive stages of thermal decomposition as well as the temperature ranges (ΔT) and mass losses (Δm) associated with these stages were appointed. Furthermore, the temperatures of DTG peaks (T_p) were also determined. Coefficients of variations of the determinations varied between 0.71 and 1.07%.

DSC scans of the studied magnesium salts were carried out with a heat-flux DSC, model 822^e (Mettler Toledo, Switzerland), with a liquid nitrogen cooling system (Dewar vessel). Samples under study, ~10 mg were accurately weighed (±0.01 mg) and encapsulated in a 40-μL flat-bottomed aluminum pans with crimped-on lids. Measurements were performed in the temperature range from 20 to 400 °C at a heating rate of 10 and 20 °C min^{-1} under an air stream at a flux rate 70 mL min^{-1}.

Calibration of calorimeter was performed by determining the heat of fusion of indium, melting point of indium was 156.60 °C, $\Delta H_f = 28.43$ J g^{-1}. Each experiment was repeated at least three times. From DSC scans, the temperatures of onset, end, peak, and the values of transition heats were determined by using the STAR^e software. Coefficients of variations of the determinations varied between 0.12 and 0.35%.

Calculations

The principal component analysis calculations were done using the *Statistica 7.1* (Statsoft®, Krakow, Poland) software. Starting point for all calculations was a matrix of the data **X** with dimensions $n \times p$, where n is a number of objects (rows) and p is a number of variables (columns). In each matrix, analyzed compounds (Mg acetate, Mg valproate, Mg lactate, Mg citrate, Mg hydrogen aspartate, Zn hydrogen aspartate) were used as the rows. Columns were the results of thermal decomposition of the studied compounds: T_o, T_e, T_p, and ΔT for the successive endothermic or exothermic peaks from the DTA curves, and T_o, T_e, ΔT, Δm, and T_p for the mass losses from the TG and DTG curves.

For PCA calculations, nine matrices **X** were constructed for data sets obtained for the first (three matrices), the second (three matrices), and for the combined data sets (three matrices) for the first and the second stage of decomposition of magnesium salts. The matrices for the first stage (dehydration) consisted of four rows, because this stage has not been observed for Mg citrate and Zn hydrogen aspartate. For the second stage of decomposition,

matrices consisted of six rows (all compounds under study). The results obtained from TG and DTG curves included 60 columns (three weighed samples, each sample heated at four heating rates, and for each heated sample five parameters were acquired—T_o, T_e, ΔT, Δm, and T_p), while the matrices prepared basing on data obtained from DTA curves consisted of 48 columns (three weighed samples, each sample heated at four heating rates, and for each heated sample four parameters were obtained—T_o, T_e, T_p, and ΔT).

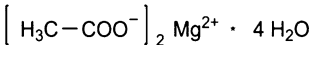
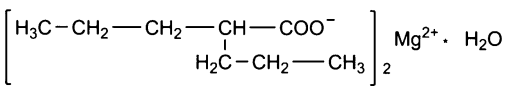
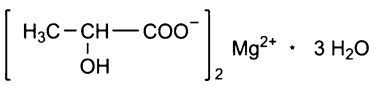
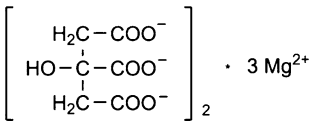
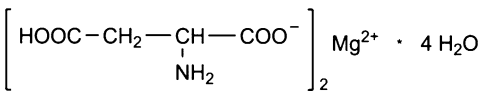
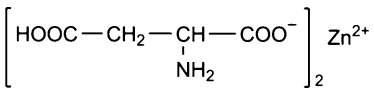
Matrix **X** was at first standardized, and then matrix **R** was calculated according to it. After further calculations, columns in matrices **P** and **W** were obtained, which were called principal components (PC). New matrix **P** reflects main relations among objects and makes possible classification of the compounds under study according to their chemical structure, whereas matrix **W** illustrates main relations among variables and enables selection of key thermal parameters, which make the best classification of the analyzed compounds.

Results and discussion

Structural formulas and basic data characterizing studied substances were set in Table 1. Structural formula indicating on the kind of chemical bonds, linear character of a basic organic structure and connected to this functional groups, as well as the molecular mass and theoretically calculated and designed from the thermograms the content of crystallization water and residue after decomposition, make easier in a some extent the determination of thermal transitions, which perhaps can occur during thermal degradation of the studied substances in conditions of linear increase of temperature.

From the analysis of the data in Table 1, it is possible to see that Mg acetate and Mg valproate are magnesium salts of monocarboxylic aliphatic organic acids, Mg lactate and Mg citrate are aliphatic hydroxyacids, containing one –OH group. More complex structures have magnesium and zinc salts of aminosuccinic acid, amino acid containing two carboxylic groups. The salt of zinc of this acid was also

Table 1 General characterization of the studied organic salts used in medicine

No.	Compound	Structural formula	Molecular formula Molar mass	Content/%[a]			
				Crystallization water		Magnesium oxide	
				Calc.	Exp.	Calc.	Exp.
1	Mg acetate		$C_4H_{14}O_8Mg$ 214.45	33.6	33.0	18.8	19.0
2	Mg valproate		$C_{16}H_{32}O_5Mg$ 328.73	5.5	4.5	12.3	13.0
3	Mg lactate		$C_6H_{16}O_9Mg$ 256.49	21.1	19.5	15.7	16.0
4	Mg citrate		$C_{12}H_{10}O_{14}Mg_3$ 451.11	–	–	26.8	26.5
5	Mg hydrogen aspartate		$C_8H_{20}O_{12}N_2Mg$ 360.56	20.0	20.5	11.2	12.0
6	Zn hydrogen aspartate		$C_8H_{12}O_8N_2Zn$ 329.58	–	–	24.7[b]	25.0

[a] Content of crystallization water and residue after decomposition were determined for 100 mg samples heated at 5 °C min^{-1} heating rate

[b] Zinc oxide was the residue after thermal decomposition of zinc hydrogen aspartate

analyzed to show the effect of a cation (Mg^{2+} and Zn^{2+}) on decomposition of a basic structure of this amino acid. It also should be mentioned that in the literature, there were no data on thermal decomposition of the studied compounds. The exception is magnesium acetate, dehydration of this salt begins at 80 °C and next anhydrous salt decomposes at 323 °C [18].

The results of DTA, TG, and DTG analyses of six studied compounds at the extreme values of sample masses and of heating rates are compiled in Table 2. As it is shown, thermal decomposition of majority salts under study occurs in two stages. The first stage of thermal destruction includes the range of temperatures (from 35 to 255 °C), in which the process of evolution of crystallization water takes place. It is exemplified by mass loss on the TG and DTG curves for four magnesium salts: Mg acetate, Mg valproate, Mg lactate, and Mg hydrogen aspartate. The DTA peaks occurred in this stage are also connected with the dehydration.

Investigated compounds differ one from another by the number of molecules of crystallization water. Analysis of thermoanalytical curves showed in Figs. 1 and 2 indicated on the fact that in the case of Mg valproate and Mg hydrogen aspartate dehydration takes place in one stage. In contrast to these compounds, dehydration of Mg acetate and Mg lactate is a process comprised of three and two

Table 2 Results of the DTA, TG, and DTG analysis of the studied compounds heated in air at different heating rates and sample masses

No.	Compound	Sample mass Heating rate	Decomposition stage	
			Temperature range of DTA peak, ΔT/°C; temperature of DTA peak, ΔT/°C Temperature range of decomposition stage, ΔT/°C; temperature of DTG peak, ΔT/°C; mass loss in TG, Δm/%	
			I	II
1	Mg acetate	50 mg 3 °C min^{-1}	40–160, 85[a]	240–370, 260[b]
			35–145, 90 (36.0)	245–340, 280 (44.0)
		200 mg 15 °C min^{-1}	45–255, 115[a]	285–530, 425[a]
			50–220, 110 (34.0)	265–425, 320 (44.5)
2	Mg valproate	50 mg 3 °C min^{-1}	65–130, 105[a]	195–445, 360[b]
			70–130, 110 (5.5)	130–380, 365 (76.0); 380–430, 385 (6.5)
		200 mg 15 °C min^{-1}	95–235, 160[a]	260–680, 580[b]
			90–190, 150 (5.0)	190–555, 510 (80.0); 555–635, 590 (3.0)
3	Mg lactate	50 mg 3 °C min^{-1}	65–155, 105[a] 60–120, 110 (16.0); 120–150, 130 (5.0)	245–450, 310[b] 250–330, 310 (31.5); 330–450, 380 (31.0)
		200 mg 15 °C min^{-1}	90–240, 150[a]	280–640, 430[b]
			60–155, 145 (16.5); 155–195, 160 (5.0)	250–390, 370 (29.5); 390–630, 410 (33.0)
4	Mg citrate	50 mg 3 °C min^{-1}		60–245, 160[a]; 245–440, 320[a]; 450–510, 475[b]
				45–210, 100 (10.5); 210–385, 320 (14.0); 380–500, 455 (49.0)
		200 mg 15 °C min^{-1}		60–265, 180[a]; 265–355, 330[a]; 355–820, 540[b]
				50–255, 165 (10.0); 255–385, 320 (13.5); 385–755, 460 (50.0)
5	Mg hydrogen aspartate	50 mg 3 °C min^{-1}	100–200, 150[a]	200–295, 220[a]; 295–375, 300[b]; 400–510, 480[b]
			120–200, 155 (20.5)	200–275, 215 (15.0); 275–375, 340 (14.5); 375–500, 485 (38.5)
		200 mg 15 °C min^{-1}	130–245, 195[a] 130–225, 185 (20.0)	245–365, 260[a]; 365–425, 405[b]; 450–860, 630[b]
				225–330, 245 (16.0); 330–440, 390 (19.0); 440–890, 520 (34.0)
6	Zn hydrogen aspartate	50 mg 3 °C min^{-1}	60–130, 85 (1.0)	140–265, 125[a]; 265–445, 435[b]
				130–250, 170 (20.5); 250–370, 340 (29.5); 370–450, 430 (24.5)
		200 mg 15 °C min^{-1}	110–160, 155 (0.5)	160–380, 240[a]; 380–660, 470[b]
				160–280, 195 (19.0); 280–420, 395 (31.0); 420–660, 465 (25.0)

The peak: [a] endothermic, [b] exothermic

substages, respectively. The proof for this is mainly the shape of peaks in DTA and DTG curves.

Very helpful in a process of investigation of dehydration of analyzed compounds appeared to be the DSC technique. DSC scans of the studied salts are show in Fig. 3, whereas the results of analysis of these curves registered at two different heating speeds are set in Table 3. Their interpretation showed that the values of dehydration temperatures are slightly increasing with the increase of heating speed. The transition heat connected with release of crystallization water takes different values for the consecutive substages of the dehydration process. The lowest amount of heat is required, when two respective moles of crystallization water is released from Mg acetate, and 1 mol of water from Mg valproate. Much more heat is needed, when 3 and 4 mol of crystallization water is released from Mg lactate and Mg hydrogen aspartate, respectively.

The analysis of shape of DSC curves confirmed that with the exception of Mg hydrogen aspartate (Fig. 3E), in each case the process of dehydration can be divided into several substages. Mg acetate release 4 mol of crystallization water in three substages (Fig. 3A), which in connection with the analysis of TG and DTG curves allows to assume (Fig. 1A) that crystallization water is released in the following sequence: 1, 1, and 2 mol. In DSC curves of Mg valproate (Fig. 3B) and Mg lactate (Fig. 3C), there are two overlapping endothermic peaks. This indicates the fact that dehydration takes place in two substages, but the analysis of TG and DTG curves (Fig. 1B, C) enables identification of two substages only for Mg lactate.

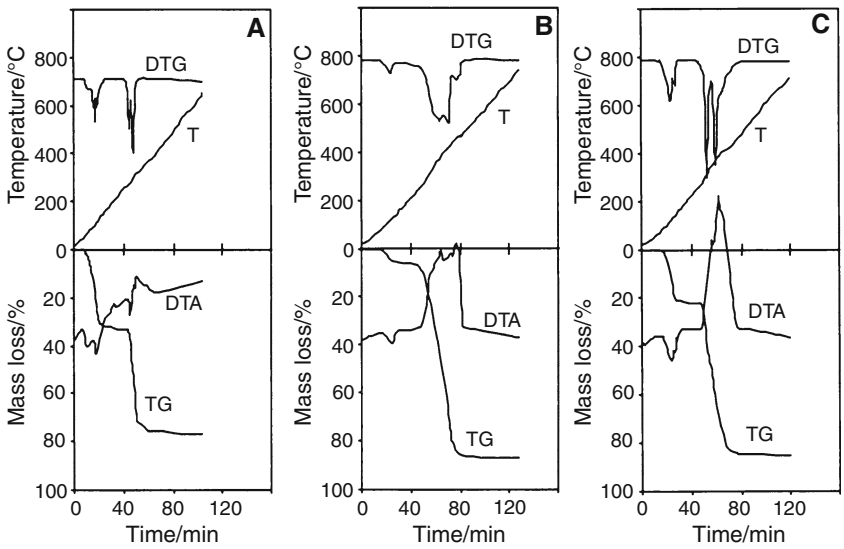


Fig. 1 DTA, TG, and DTG curves of the thermal decomposition of: **A** magnesium acetate, **B** magnesium valproate, and **C** magnesium lactate. Hundred milligram samples were heated at 5 °C min^{-1} heating rate

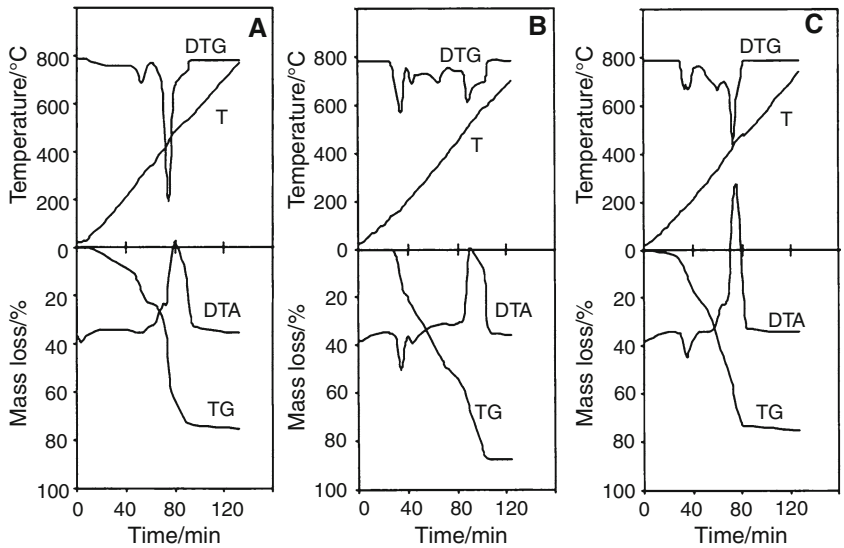


Fig. 2 DTA, TG, and DTG curves of the thermal decomposition of: **A** magnesium citrate, **B** magnesium hydrogen aspartate, and **C** zinc hydrogen aspartate. Hundred milligram samples were heated at 5 °C min^{-1} heating rate

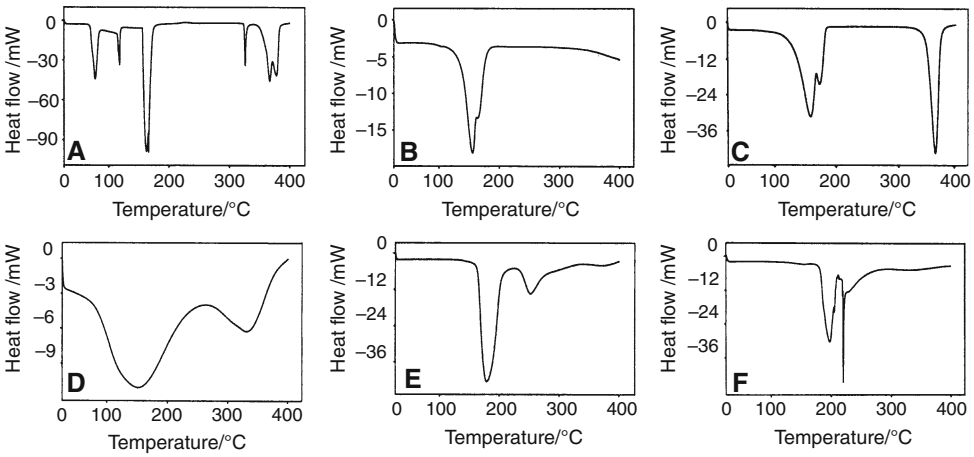


Fig. 3 DSC scans of the studied compounds: **A** magnesium acetate, **B** magnesium valproate, **C** magnesium lactate, **D** magnesium citrate, **E** magnesium hydrogen aspartate, and **F** zinc hydrogen aspartate. Ten milligram samples were heated at a rate of 10 °C min^{-1}

Table 3 Results of the DSC analysis of the compounds being dehydrated in air at different heating rates

No.	Compound	Sample mass Heating rate	Dehydration/°C	Onset/°C	Endset/°C	Transition heat/J g^{-1}	Enthalpy/kJ mol^{-1}
1	Mg acetate	10.41 mg	75.84	69.31	83.23	–187.38	3.38
		10 °C min^{-1}	116.81	114.20	118.91	–41.86	0.75
			161.29	155.59	177.40	–578.80	20.85
		10.45 mg	75.64	69.87	84.11	–175.08	3.15
		20 °C min^{-1}	116.49	114.95	119.70	–18.90	0.34
			156.00	152.54	175.53	–581.14	20.94
2	Mg valproate	10.08 mg	154.67	136.00	166.44	–233.54	4.21
		10 °C min^{-1}					
		10.24 mg	162.59	143.03	190.21	–223.37	4.02
		20 °C min^{-1}					
3	Mg lactate	10.32 mg	157.69	131.74	172.69	–572.58	30.95
		10 °C min^{-1}					
		10.35 mg	165.90	143.21	198.22	–574.21	31.03
		20 °C min^{-1}					
4	Mg hydrogen aspartate	10.40 mg	176.23	163.45	202.34	–602.31	43.40
		10 °C min^{-1}					
		10.27 mg	180.75	164.82	214.24	–600.80	43.29
		20 °C min^{-1}					

Anhydrous compounds are totally thermally decomposed in the second stage of destruction (Figs. 1, 2). The reactions of decomposition of organic structure, which take place in a solid phase in conditions of the constant temperature increase of the system, have multidirectional course. The proof for this is the shape of TG and DTG curves indicates that the decomposition of a particular compound goes through several substages. However, it is not possible to accurately separate the following substages of thermal decomposition and identification of intermediate products occurring in these conditions. They are finally being decomposed, which process is connected with complete burning of charred residue, and MgO or ZnO are appearing as the final products of decomposition. The overall thermal effect of this stage is exothermic, which is confirmed by the large peak in DTA curve. The presence of Mg^{2+} in the residue after decomposition was confirmed by the reaction with Magnezone (4-nitrobenzeno-azo-rezorcine), whereas Zn^{2+} was found based on the reaction with tetrathiocyanatemercure(II) ammonium [19].

The principal component analysis was used for complex interpretation of the thermoanalytical data sets for studied compounds [20, 21]. This method greatly makes easier interpretation of multidimensional databases by reduction

of multidimensionality and by clear way of presentation of the results. However, these calculations are performed at the cost of the loss of information, because PCA does not takes into consideration all variability within experimental data, but it allows to identify some factors, which describe the crucial part of variability in a data matrix. As the result of performed calculations, two principal components were obtained, of which eigenvalues were higher than 1. Two first principal components explained more than 90% of variability for most matrices. However, in the case of two matrices obtained from TG and DTG curves for the second stage of decomposition, two first principal components explained 89% of variability.

Relations between chemical structure of the investigated compounds and the results of analysis of DTA, TG, and DTG curves of their thermal decomposition were shown graphically in Fig. 4A (stage I, dehydratation) and Fig. 4B (stage II, decomposition).

In the first stage, the studied salts release crystallization water, and this process is accompanied by endothermic peak in DTA curve and several- or several tens of percent loss of mass in TG and DTG curves. Analyzing the distribution of these salts in two-dimensional space, it was found that on the left side of the plot PC1 versus PC2 (Fig. 4A), in similar range of values of PC1, there are three compounds located: Mg acetate, Mg valproate, and Mg lactate. Mutual element in common for these substances is the fact that they are aliphatic acids containing single group –COOH, and they do not have a heteroatom. Presence of hydroxyl group in the molecule is the reason for moving of Mg lactate toward positive values in PC1 axis.

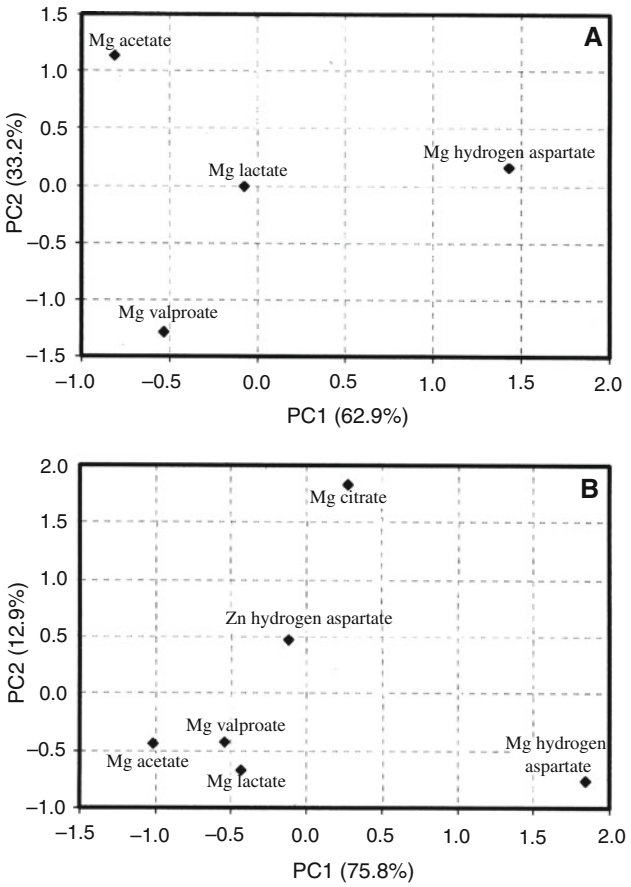


Fig. 4 Scatter plots of the first two principal component vectors (PC1 vs. PC2) for the data set acquired from DTA, TG and DTG curves for: **A** the first stage (dehydration) and **B** the second stage (decomposition) of the analyzed compounds

Taking into consideration the distribution of these compounds along PC2 axis, it is possible to state that certain impact on their location has the molecular mass. Higher the molecular mass, then given magnesium salt is closer to lower values of PC2. For example, Mg acetate with the lowest molecular mass is described by the highest value on PC2 axis (about 1.2), whereas Mg valproate with the highest molecular mass can be found in the region of negative values on PC2 axis (about −1.3).

The fourth compound containing crystallization water, Mg hydrogen aspartate, differs from the other magnesium salts with that it has amine group and two groups –COOH. This crucial difference causes that in the two-dimensional diagram of PC1 versus PC2, this compound is on the right side of the plot, on the opposite side in relation to the other hydrates, in positive range of PC1 values.

Anhydrous compounds have their thermal decomposition in the second stage with the formation of the residue, which is oxide of particular cation (Mg^{2+} or Zn^{2+}). This disintegration is accompanied by several tens percent mass loss and large exothermic peak in DTA curve. Graphical interpretation of PCA calculations for six investigated salts based on the data obtained from DTA, TG, and DTG curves for this stage of decomposition is shown in Fig. 4B. The obtained results indicate the fact that similar to that was shown in Fig. 4A, in close range of values of PC1 and PC2 there are the same three compounds: Mg acetate, Mg valproate, and Mg lactate. The other substances (Mg citrate, Mg hydrogen aspartate, Zn hydrogen aspartate) are located in two-dimensional plot in different, separate one from another position. This can be explained by the lack of similarity in their chemical structure. Mg citrate is tricarboxylic acid, whereas location of these two salts of aminosuccinic acid indicates the fact that the crucial influence on decomposition of the basic structure of this acid has cation of the metal, with which the salt is formed.

The principal component analysis calculation was also performed for the matrices obtained by joining of the thermoanalytical data acquired for the first and the second stages, however, by joining of two stages of decomposition it is possible to analyze only the relations among four substances (Mg acetate, Mg valproate, Mg lactate, and Mg hydrogen aspartate). The obtained results do not indicate

the relations between the studied compounds other than showed in Fig. 4A.

Conclusions

As the effect of the conducted studies, the general scheme of decomposition of the investigated compounds was proposed, in which two basic stages of decomposition were separated. On this basis, the ranges of temperature were designed, in which the analyzed substances can be technologically processed without loss of their specific physicochemical properties.

The results of PCA showed that the course of thermal decomposition of the studied salts depends in certain degree on their chemical structure. This relation is best analyzed in two-dimensional space of PC1 versus PC2. Two first principal components explain together 90% of variance. Slightly higher values of variance were obtained for the matrix created on the basis of analysis of DTA curves then in the case of TG and DTG. The joining of the results of DTA with TG and DTG, or the first with the second stage of decomposition, did not result in significant difference in the explained variance.

The graphical interpretation of PCA results has revealed that points on PC1 versus PC2 diagrams corresponding to the compounds of similar chemical constitution are localized in the similar ranges of the first two PC's values. This proves that thermal decomposition reflects similarity in the structure of magnesium salts of organic acids.

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