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Synthesis and Investigation of Magnesium Stearate with Coordinated Compounds with Acetamide and Nicotineamide

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Author's contribution

The sole author designed, analyzed, interpreted and prepared the manuscript.

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ABSTRACT

Homogenous coordination compounds of magnesium stearate with acetamide and nicotinamide were synthesized. Composition, individuality, methods of acetamide, nicotinamide molecules and stearate fragments coordination of coordinated compounds were established. In addition, thermal behavior of produced complexes was studied. IR-spectrum of absorption and X-ray phase analysis methods were used to prove of formation of chemical bonds and thermolysis for the definition of composition and individuality of newly synthesized compounds.

Keywords: Complex compounds; synthesis; composition; analysis methods; IR-spectrum of absorption; X-ray phase analysis; thermolysis.

1. INTRODUCTION

It's known synthesis of new compounds with effective and useful properties for agricultural aims is one of the actual problems of contemporary chemistry. At the present time complex compounds of s-, p-, d-metals with specific properties are being applied successfully in broad spectrum.

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In the current time an important issue of the chemistry of coordination compounds is the exploration of various properties of preparations synthesized from physiologically active substances that are amide derivatives with macrometal ions, including synthesis, study of the structure and properties of complex compounds [1,2]. Practical value of the field opens great possibilities for the national economy. The investigation of coordination compounds is consistent not only with the purpose of expanding the areas of their practical application, but also in solving the fundamental problems of chemical science, including questions linked to the nature of the chemical bond and structure [2,3].

In Refs. [4-8]. coordination compounds of formates, acetates, oleates, stearates, benzoates and metal nicotinates with acid amides were synthesized and studied. In the literature there are no data on acetamide, urea and thiourea complex compounds of magnesium palmitate. Due to above mentioned problems, current work presents synthesis and investigation of properties of coordinated compounds of magnesium stearate with acetamide and nicotineamide.

2. MATERIALS AND METHODS

For the fulfilling of the work magnesium palmitate (chemical pure), acetamide and nicotineamid (chemical pure for analysis) were obtained from local sources.

The synthesis was carried out mechanochemically, by trituration of a mixture of magnesium stearate: acetamide in a molar ratio of 1 : 2 for 20 minutes at room temperature in a ball mill with working bodies 2-5 (1.0 liter mill volume) [7]. All mixed-amide complexes of magnesium with amides were obtained by the above method. The complex compound of the composition $Mg(C_{17}H_{35}COO)_2 \cdot 2CH_3CONH_2 \cdot 4H_2O$ was synthesized by intensive stirring of 0.6273 g (0.001 mol) of magnesium stearate dihydrate with 0.1181 g (0.002 mole) of acetamide in a ball mill at room temperature for 0.15 hours.

In the synthesis of the complex compound of the composition $Mg(C_{17}H_{35}COO)_2 \cdot 2NC_5H_4CONH_2 \cdot 2.5H_2O$, 0.6273 g (0.001 mole) of magnesium stearate dihydrate was mixed with 0.2445 g (0.002 mole) of nicotinamide in a ball mill at room temperature for duration 0.15 hours.

Analysis of synthesized compounds for magnesium content was carried out according to Ref 7. Nitrogen was determined by the Dumas method [9], carbon and hydrogen by burning in an oxygen flow (Table 1). To determine the individuality of synthesized compounds, radiographs were taken on a DRON-2.0 unit with a Cu-anticathode [10]. To calculate the interplanar distances, the tables [11,12] were used, and the relative intensity of the I / I1 line was determined as a percentage of the most pronounced reflex at the maximum. IR absorption spectra were recorded in the 400-4000 cm^{-1} region on the AVATAR-360 spectrometer of Nicolet.

The thermal analysis was carried out on the F.Paulik-J.Paulik-L.Erdey system [12] derivatograph at a rate of 9 deg / min and a sample of 0.076-0.094 g with a sensitivity of T-900, TG-200, DTA, DTG-1 / 10. Zaris was carried out under atmospheric conditions. The holder was a platinum crucible with a diameter of 10 mm without a cover. Al_2O_3 was used as a standard.

3. RESULTS AND DISCUSSION

Table 1 summarized the results below the results of element analysis of homogeneous coordination compounds of magnesium stearate to definy Mg and N.

Table 1. Results of elemental analysis of homogeneous coordination compounds of magnesium stearate with acetamide and nicotinamide

Compounds	Mg,%		N,%		C,%		H,%	
	Found	Counted	Found	Counted	Found	Counted	Found	Counted
$Mg(C_{17}H_{35}COO)_2 \cdot 2CH_3CONH_2 \cdot 2H_2O$	3.30	3.26	3.79	3.76	64.70	64.45	11.47	11.36
$Mg(C_{17}H_{35}COO)_2 \cdot 2NC_5H_4CONH_2 \cdot 2.5H_2O$	2.73	2.76	6.29	6.36	65.71	65.47	10.01	9.96

Comparison of the interplanar distances and relative intensities of magnesium stearate, acetamide, nicotinamide and new complex compounds of the compositions $Mg(C_{17}H_{35}COO)_2 \cdot 2CH_3CONH_2 \cdot 2H_2O$, $Mg(C_{17}H_{35}COO)_2 \cdot 2NC_5H_4CONH_2 \cdot 2.5H_2O$ showed that they differ significantly from each other, from similar and the original starting compounds. Consequently, the synthesized complexes of magnesium stearate have individual crystal lattice (Table 2).

IR spectra of free molecules of ligands and synthesized compounds were studied.

In the IR absorption spectrum of acetamide (AA), frequencies (cm^{-1}) were found at 3387-ν(NH_2), 3194 - 2δ(NH_2), 1670 - ν(C=O), 1626 - δ(NH_2), ν(CO), 1395 - ν(CN), 1348 - δ(CH_3), 1154 - ρ (NH_2), 1048 - ρ(CH_3), 1005 - ν(C-C), 875 - ν(C-C), 582 - δ(NCO) and 464 - δ(CCN).

Table 2. Interplanar distances and relative intensities of lines of homogeneous complex compounds of magnesium stearate

Compounds	d,Å	I,%	d,Å	I,%	d,Å	I,%	d,Å	I,%	d,Å	I,%
$Mg(C_{17}H_{35}COO)_2 \cdot$	15.73	2	4.82	3	2.56	1	1.967	2	1.555	1
$\cdot 2CH_3CONH_2 \cdot 2H_2O$	14.69	2	4.77	3	2.55	1	1.948	1	1.500	1
	13.52	2	4.49	3	2.49	1	1.881	2	1.492	1
	12.59	2	4.31	3	2.42	6	1.868	2	1.469	1
	10.55	6	4.22	3	2.36	1	1.847	1	1.461	1
	9.71	1	4.06	100	2.31	3	1.792	1	1.451	1
	9.00	1	3.98	12	2.29	2	1.773	1	1.429	1
	8.78	1	3.79	40	2.26	1	1.760	1	1.419	1
	7.40	1	3.51	12	2.23	1	1.739	1	1.396	1
	6.68	1	3.28	2	2.16	1	1.724	1	1.378	1
	6.53	2	3.10	1	2.14	2	1.694	2	1.363	1
	5.95	5	3.07	1	2.12	2	1.693	1	1.355	1
	5.74	3	2.97	10	2.08	2	1.665	1	1.332	1
	5.52	3	2.90	4	2.06	2	1.648	1	1.311	1
	5.40	2	2.86	3	2.03	2	1.637	1	1.305	1
	5.27	3	2.77	1	2.02	2	1.611	1	1.302	1
	5.25	4	2.73	1	2.00	2	1.606	1		
	5.04	3	2.61	1	1.977	2	1.577	1		
$Mg(C_{17}H_{35}COO)_2 \cdot$	16.44	2	4.66	3	2.74	2	1.948	1	1.488	1
$\cdot 2NC_5H_4CONH_2 \cdot 2.5H_2O$	15.52	3	4.38	74	2.71	3	1.877	2	1.467	1
	14.69	3	4.26	8	2.69	3	1.854	1	1.453	1
	12.59	2	4.17	3	2.63	2	1.832	1	1.447	1
	12.17	2	4.07	7	2.61		1.791	2	1.440	1
	10.55	2	3.99	11	2.57	2	1.754	2	1.431	1
	10.06	2	3.92	7	2.48	2	1.723	2	1.413	1
	9.19	2	3.86	11	2.40	8	1.702	1	1.405	1
	8.36	3	3.70	100	2.33	2	1.693	1	1.393	1
	7.99	2	3.62	5	2.30	2	1.659	1	1.390	1
	7.53	2	3.53	11	2.28	2	1.619	1	1.378	1
	7.28	3	3.45	16	2.25	4	1.616	1	1.370	1
	6.83	2	3.40	25	2.20	1	1.594	1	1.361	1
	6.38	7	3.30	2	2.16	11	1.584	1	1.357	1
	5.83	30	3.22	26	2.14	2	1.547	1	1.340	1
	5.52	3	3.08	3	2.12	1	1.527	1	1.323	1
	5.46	3	2.99	9	2.10	2	1.520	1	1.315	1
	5.16	5	2.91	5	2.06	2	1.514	2		
	4.99	3	2.85	1	2.02	2	1.509	1		
	4.85	3	2.80	2	1.98	1	1.497	1		

Table 3. The values of the characteristic frequencies (cm^{-1}) in the IR absorption spectra of a free molecule of acetamide and nicotinamide and their coordination compounds with magnesium stearate

Compound	ν(C=O)	ν(C=N)	νk, δ(C=O)	$\nu_{as}(COO^-)$, $\nu_s(COO^-)$
CH_3CONH_2	1670	1390		
$NC_5H_4CONH_2$	1681		1593, 1029, 703	
$Mg(C_{17}H_{35}COO)_2 \cdot 2CH_3CONH_2 \cdot 4H_2O$	1665	1414		1542, 1473
$Mg(C_{17}H_{35}COO)_2 \cdot 2NC_5H_4CONH_2 \cdot 2.5H_2O$			1619, 1684, 718	1562, 1415

The IR spectrum of the uncoordinated nicotinamide molecule (ANC) has frequencies at 3366 - $\nu(NH_2)$, 3161 - $2\delta(NH_2)$, 3060 - ν(CH), 1680 - ν(C=O), 1619 - $\delta(NH_2)$, 1594 - $\nu_к$, 1574 - $\nu_к$, 1483, 1423 - $\nu_к$, δ(CCN), 1398,1342 - ν(CH), δ(CCN), 1200 - δ(CCN), 1143,1127 - $\nu(NH)_2$, δ(CCN), 1086 - δ(CCN), ν(CO), $\nu_к$, 1029 - $\nu_к$, δ(CCN), 986 - ν(CC), 831 - ν(CC), δ(CCC), 777,703 - δ(CCN), δ (CO), 624,604 - δ(CO), δ(CNC), 514 - δ(CO), δ(CCC).

In the IR absorption spectrum of the free molecule of acetamide, two frequencies at 1670 and 1390 cm^{-1} correspond primarily to the valence vibration of the C = O and C-N bonds. With the transition to a coordinated state, the C = 0 frequency decreases by 5 cm^{-1}, while the C-N frequency increases by 24 cm^{-1}. Such a change in frequencies indicates the coordination of the acetamide molecules with the magnesium ion through the oxygen atom of the carbonyl group [13].

In the IR absorption spectrum of the nicotinamide molecule, there is a sufficient number of frequencies and the frequency of ν (ring) is observed at 1593 cm^{-1}, which in the case of the complex is significantly increased. The absorption bands at 1029-νk and 703 cm^{-1} (CNN) belong to the vibrations of the ring. These changes may be evidence of the coordination of nicotinamide with the magnesium ion through the nitrogen heteroatom of the pyridine ring (Table 3).

The frequency difference $\nu_{as}(COO) - \nu_s(COO)$ in the complexes of acetamide and nicotinamide is 69 and 147 cm^{-1}, which is characteristic for the bidentate coordination of the stearate group. Thus, complex compounds have six coordinative sites of the magnesium ion (Figs. 1. 2).

The thermal behavior of synthesized compounds was established by the method of derivational analysis [14-16]. Intermediate thermolysis products were obtained and the composition of the compounds was established.

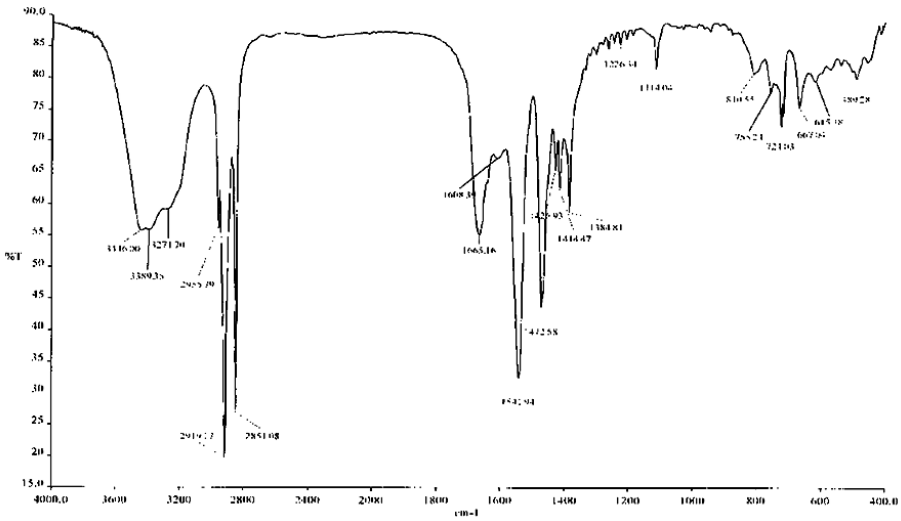

Fig. 1. IR absorption spectrum of a homogeneous complex compound of magnesium stearate with an acetamide of the composition $Mg(C_{17}H_{35}COO)_2 \cdot 2CH_3CONH_2 \cdot 4H_2O$

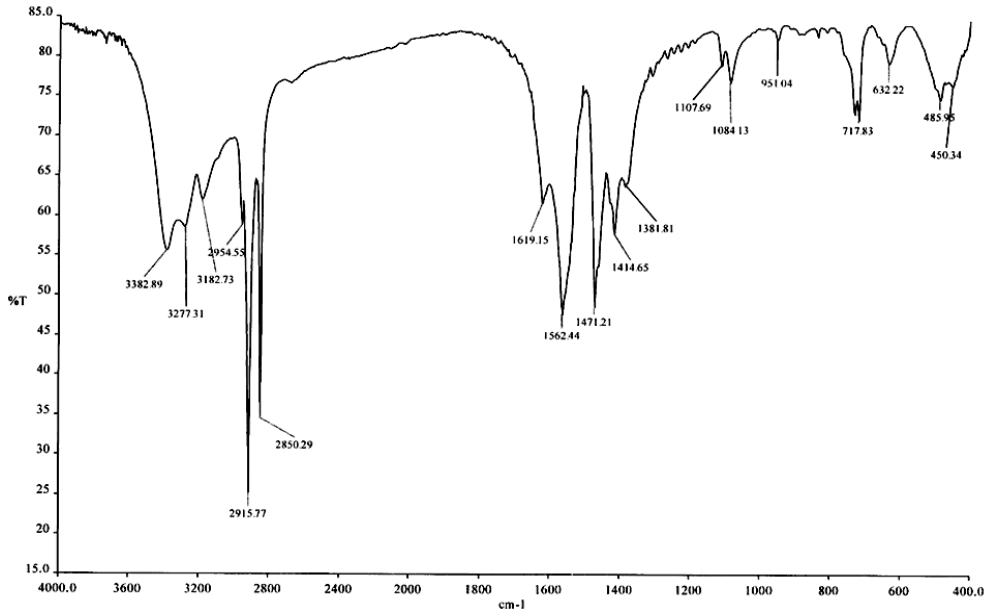

Fig. 2. IR absorption spectrum of a homogeneous complex compound of magnesium stearate with nicotinamide of the composition $Mg(C_{17}H_{35}COO)_2 \cdot 2NC_5H_4CONH_2 \cdot 2.5H_2O$

The heating curve of the complex compound $Mg(C_{17}H_{35}COO)_2 \cdot 2CH_3CONH_2 \cdot 4H_2O$ revealed six endothermic effects at 80, 125, 190, 235, 238, 331, 352°C and nine exothermic effects at 300, 376, 395, 458, 505, 580, 648 , 697 and 780 °C. The appearance of the first and second endothermic effect is due to the removal of four water molecules. The mass loss in the temperature range at 60-142°C is 9.22%.

The nature of the subsequent thermal effects is accompanied by a stepwise decomposition of the anhydrous compound. In the temperature ranges 142-210, 210-248, 248-320, 320-340, 340-364, 364-382, 382-420, 420-460, 460-526, 526-600, 600-675, 675-722, 722-900^{o}C loss of weight is 5.92; 5.79; 3.95; 2.63; 2.89; 3.68; 2.63; 40.13; 15.79; 0.12; 0.13; 0.61; 0.13%. The total mass loss in the temperature range 60-900°C on the TG curve is 93.82%.

Four endothermic effects at 115, 150, 678, 858°C and thirteen exothermic effects at 178, 240, 282, 348, 378, 392, 444, 469, 529 were found on the heating curve of the complex compound $Mg(C_{17}H_{35}COO)_2 \cdot 2NC_5H_4CONH_2 \cdot 2{,}5H_2O$, 567, 634, 743 and 791°C.

The appearance of the first endothermic effect is due to the removal of two halves of the water molecule. The mass loss in the temperature range 80-127°C is 5.11%. The nature of the subsequent thermal effects is accompanied by a stepwise decomposition of the anhydrous compound. In the temperature ranges 127-164, 164-202, 202-255, 255-310, 310-370, 370-380, 380-425, 425-450, 450-473, 473-540, 540-620, 620-640 , 640-720, 720-760, 760-815, 815-870 Co loss of weight is 0.21; 0.85; 5.11; 15.47; 15.57; 1.06; 19.02; 12.70; 8.98; 7.41; 1.49; 0.21; 2.23; 0.10; 0.11; 0.11%. The total mass loss in the temperature range 80-870°C on the TG curve is 95.74%.

4. CONCLUSION

It was synthesized coordinated compounds of magnesium stearate with acetamide and nicotineamide that can be applied as a biological stimulator for cultivars. It's important to note that obtained compounds were synthesized by the use of mechanochemical methods which don't require expensive chemicals. As we know it's important for scale up processes. The optimal conditions of synthesis and ratio of synthesized compounds for application were established. Besides these, composition, individuality and thermal property of coordinated compounds were explored and proved.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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