

KINETICS OF THERMAL DESTRUCTION OF ALUMINUM OXYHYDRATES OBTAINED IN THE PRESENCE OF SURFACE-ACTIVE SUBSTANCES

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Abstract

The calculation and analysis of the kinetic parameters of the thermal decomposition of aluminum oxyhydrates obtained by the hydrolysis of aluminum nitrate in the presence of a non-inogenous surface-active substance (surfactant) - isooctyl ether of decaethylene glycol (IEDG) is presented. For comparison, samples of pure aluminum oxyhydrates were synthesized without the use of surfactants.

Thermogravimetric measurements were performed simultaneous thermal analyzer Netzsch STA 449 F3 Jupiter. As a result of the experiment for each sample, we obtained graphs of the TG/DTG mass loss in the temperature range from 40⁰C to 1260⁰C with marking in sections and the corresponding tabular data.

The processing of tabular data of the corresponding thermograms recorded in increments of 0,5 degrees was carried out in two stages. At the first stage, according to the dependences of the form $\log[-\log(1-\Delta m/\Delta m_{\max})]$ from $10^3/T$, linear temperature ranges were determined. Each stage of thermolysis was processed according to the $\log[-\log(1-\alpha)]$ graph from $10^3/T$ [1]. The reliability of using the calculated trend model for describing experimentally observed dependencies was verified using the Fisher criterion. The apparent activation energy (E_a) and the value of the parameter (n) characterizing the appearance and development of crystallization centers are determined graphically. To find the rate constant (k), the Johanson – Melya – Arami – Yerofeyev – Kolmogorov equation was used [2].

At the second stage of processing the results of thermolysis, the Freeman–Carroll method was used [3]. TG/DTG curves are processed by temperature differentiation from the beginning to the end of the destruction process over the entire data array. As a result, characteristic peaks of mass loss are determined. According to this algorithm, the calculation and analysis of the values of the kinetic parameters describing the sequence and patterns of thermal transformations of aluminum oxyhydrates depending on the IEDG content during their synthesis were carried out.

Key words: *thermogravimetry, thermolysis, kinetic parameters, aluminum oxyhydrates, isooctyl ether of decaethylene glycol, surfactant.*

Introduction

Aluminum oxyhydrates and their thermal decomposition products are widely used in science and technology. Of particular practical importance are nanomaterials with predetermined functional properties obtained on the basis of aluminum oxides and hydroxides. An important role in determining the scope of such materials is played by an experimental study of transformations occurring under conditions of programmed temperature change. Thermogravimetry in non-isothermal conditions is one of the most commonly used methods in the study of thermal destruction of oxide nanomaterials. At the same time, the study of the thermal decomposition of aluminum oxyhydrates synthesized by new methods, including using surfactants, remains relevant.

Thermal decomposition of a substance with the participation of solid phases refers to complex heterogeneous processes involving several stages: the actual chemical reaction; rearrangement of the lattice of the initial phase into a new one; diffusion of gases in the capillaries of a solid; transfer of gaseous products and their release; crystallization of the solid reaction product [1, 4]. Depending on the speed of a particular stage, the total reaction rate can be determined by chemical, diffusion, or crystallization processes. Reactions of the $A_s \leftrightarrow B_s + S_g$ type, in which the chemical interaction is not complicated by diffusion processes, are topochemical. They are characterized by an increase in the reaction rate at the initial moments of the process and the achievement of the limiting value, followed by a gradual decrease to zero. An external sign of topochemical reactions is the S-shaped kinetic curve [5], (Fig. 1).

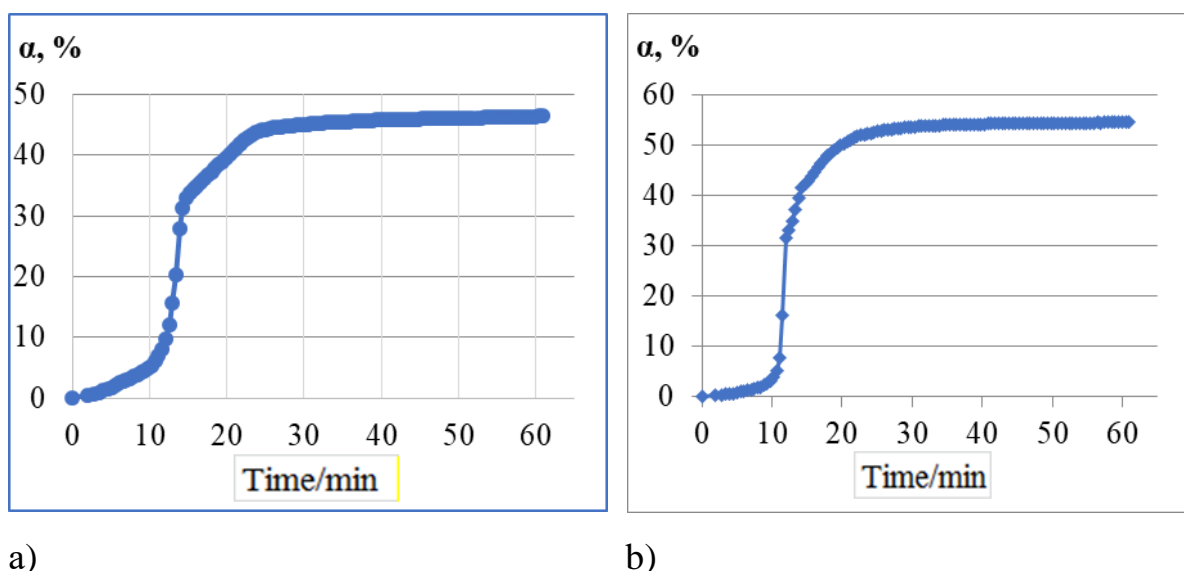


Figure 1. Dependence of the degree of conversion during thermolysis of aluminum oxyhydrates formed without IEDG (a) and in the presence of 20% IEDG (b).

Such processes do not begin on the entire surface of the initial solid, but on individual points, the so-called potential reaction centers, from where the process gradually spreads deeper [5].

In this regard, the preferred model for calculating the kinetic parameters of the thermolysis of aluminum oxyhydrates is the nucleation model, which is based on the assumption that the limiting stage of solid-phase interaction is the formation of new phase nuclei or their growth. The speed of such a process is determined not only by chemical, but also by crystallochemical factors [4].

It should be noted that during thermal analysis proceeding under non-isothermal conditions, a number of assumptions are confirmed by numerous experimental data: the transformation is described by the Arrhenius equation; diffusion of reaction products does not significantly affect the temperature field of the sample, since weighed samples of less than 1 g and moderately high temperatures [1].

The aim of this work is to study the features of the nonisothermal kinetics of thermal decomposition of aluminum oxyhydrates obtained by hydrolysis of aluminum nitrate in the presence of IEDG. Determination by calculation methods of the main kinetic parameters of the process and identification of the dependence of their changes under the influence of various surfactant contents.

Experiment

Samples of aluminum oxyhydrates were synthesized as follows. Aluminum nitrate 9-water qualification "chemically pure" dissolved in distilled water to obtain a 37,5 wt.% aqueous solution, to which was added 1, 5, 10 and 20% of IEDG, known in production as an auxiliary substance OP-10 [6]. After that, it was kept for 1,5 hours at 80°C until the components were completely dissolved and 2 hours at room temperature. Aliquots of the resulting compositions with 0, 1, 5, 10, and 20% IEDG, respectively, with a volume of 10 ml, were placed in a reactor with a vessel containing a 10 wt.% ammonia solution and left to form a complete precipitate of aluminum oxyhydrate for 20 days. The precipitated solid phase was washed in 96 wt.% ethanol for 4 days, then dried at 80°C for 2 days.

Thermogravimetric measurements were performed simultaneous thermal analyzer Netzsch STA 449 F3 Jupiter. The shooting conditions were as follows: heating rate 20 K/min; temperature range from 40⁰C to 1260⁰C; atmosphere - air; crucible material is corundum. Weights of samples from 17 to 21 mg.

Results

As a result of this study, for each sample, we obtained graphs of the TG/DTG mass loss with marking in sections (Fig. 2) and the corresponding tabular data.

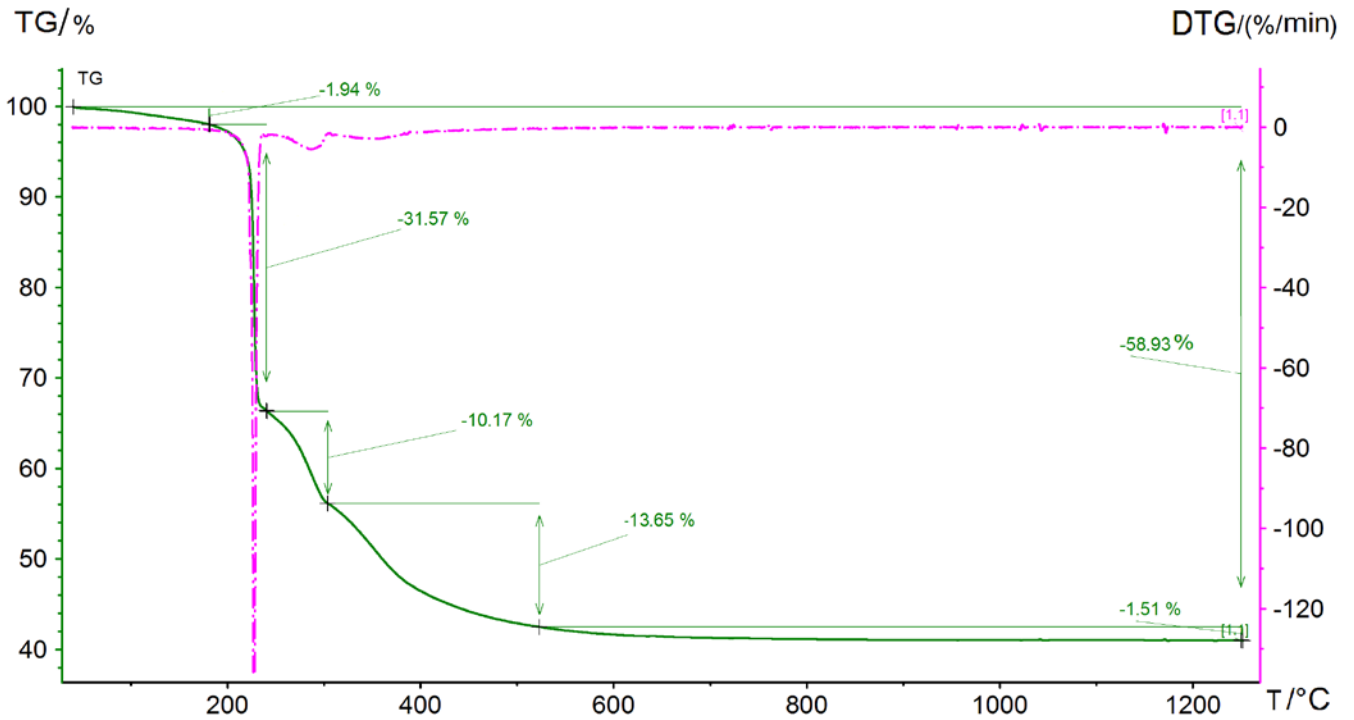


Figure 2. TG/DTG curve for pure aluminum oxyhydrate in the original software format

The tabular data of the corresponding thermograms recorded in increments of 0,5 degrees were processed in two stages. At the first stage [1], linear temperature intervals were determined by the dependences of the form $\Delta = \log[-\log(1 - \Delta m / \Delta m_{\max})]$ on $10^3/T$, where Δm is the mass loss at temperature T ; Δm_{\max} is the maximum mass loss sample; T , K - temperature (Fig. 3). According to this algorithm, the number of stages and temperature intervals of the process of thermal destruction of all the studied samples were determined.

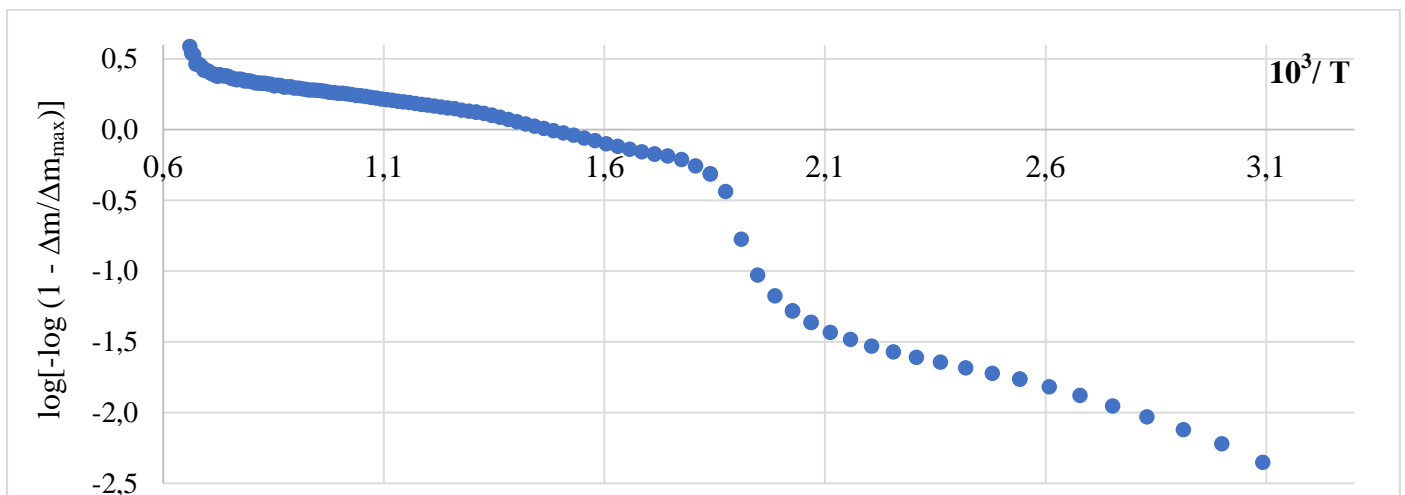


Figure 3. The dependence of $\Delta = \log[-\log(1 - \Delta m / \Delta m_{\max})]$ on $10^3/T$ for pure aluminum oxyhydrate.

Each stage of thermolysis was processed according to the dependencies $\Delta = \log[-\log(1 - \alpha)]$ on $10^3/T$ [1], (Fig. 4).

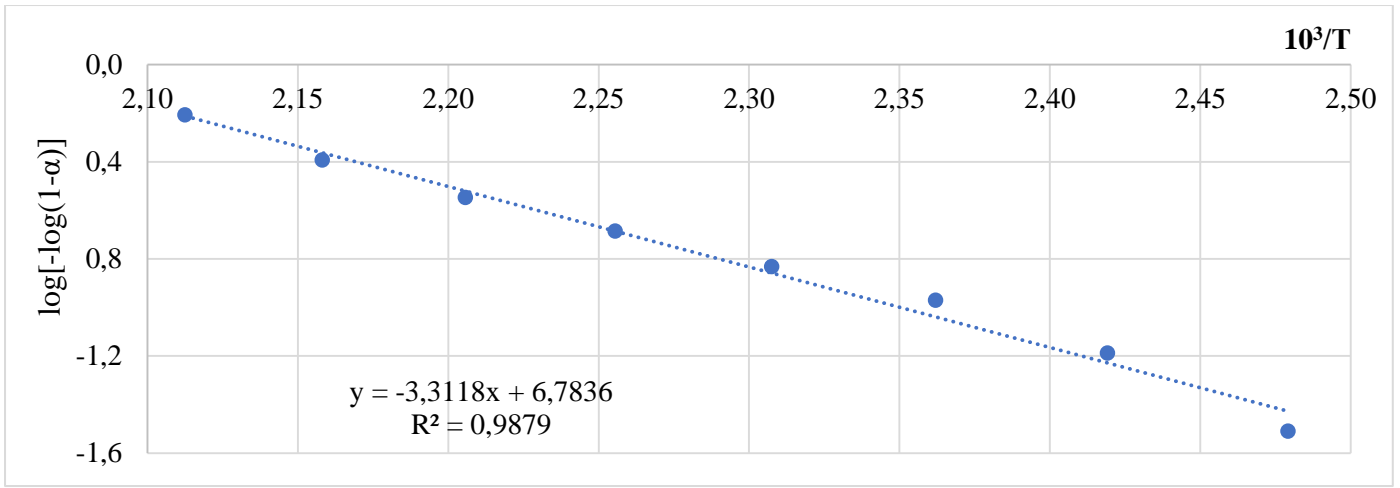


Figure 4. The dependence of $\Delta = \log[-\log(1-\alpha)]$ on $10^3/T$ in the second stage of thermolysis of aluminum oxyhydrate obtained in the presence of 5% IEDG.

The reliability of using the calculated trend model to describe the experimental data was checked using the Fisher criterion for the values of the reliability coefficient of approximation R^2 of the obtained linear dependences of $\log[-\log(1-\alpha)]$ on $10^3/T$ (Fig. 4). The calculated value of the F-criterion (F_{cal}) was found by the formula (1):

$$F = \frac{(n-l-1) \cdot R^2}{m(1-R^2)} \quad (1),$$

where R^2 is the approximation confidence coefficient, n is the number of measurements, l is the number of factors taken into account in the model.

The critical value of the F-criterion (F_{cr}) was determined at a significance level of 0,05 [7]. The decision on the possibility of using this algorithm for calculating the kinetic parameters was made after confirming compliance with the conditions $F_{cal} > F_{cr}$.

The apparent activation energy (E_a), the value of the parameter (n) characterizing the appearance and development of crystallization centers, was calculated graphically.

To find the constant k , we used the equation (2) of Johanson – Melya – Arami – Yerofeyev – Kolmogorov [2]:

$$\lg[-\lg(1-\alpha)] = \lg k + n \lg t \quad (2),$$

where α is the proportion of reacted substance; k is the rate constant; t is the decomposition time; n is a parameter characterizing the emergence and development of crystallization centers.

The value of k was used to find another rate constant K (dimension [t⁻¹]) through the Sakovich relation (3) [1; 2]:

$$K=k^{1/n} \cdot n \quad (3).$$

The results of calculating the kinetic parameters according to the thermal analysis of aluminum oxyhydrates according to the procedure [1], taking into account the reliability assessment of the applied trend model, are presented in the summary table 1.

Table 1 - Kinetic parameters calculated by the selected stages of thermolysis.

Thermolysis stages, parameters	Sample No. _% IEDG									
	1.1_0%	1.2_0%	2.1_1%	2.2_1%	3.1_5%	3.2_5%	4.1_10%	4.2_10%	5.1_20%	5.2_20%
1, °C	39-110	39-110	28-98	39-110	39-110	39-120	40-110	40-120	40-120	40-120
R²	0,9943	0,9954	0,9964	0,9914	0,9873	0,9936	0,9956	0,9818	0,9842	0,9836
F_{cal}>F_{cr}	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
E_a, kJ/mol	15,28	17,02	17,19	15,73	14,08	14,24	13,75	13,28	15,60	14,76
n	1,84	2,05	2,07	1,89	1,70	1,71	1,66	1,60	1,88	1,78
K, min⁻¹	0,28	0,34	0,51	0,32	0,25	0,28	0,27	0,22	0,27	0,25
Δ weight, %	2,6	1,5	6,2	1,7	1,7	2,0	1,4	1,1	1,1	1,0
2, °C	120—210	120-220	98-218	120-210	120-190	120-210	110-200	120-190	120_190	120-190
R²	0,9888	0,9869	0,9785	0,9836	0,9925	0,9879	0,9851	0,9907	0,9908	0,9841
F_{cal}>F_{cr}	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
E_a, kJ/mol	24,67	23,30	15,77	22,99	31,96	27,52	26,48	33,71	32,72	32,33
n	2,97	2,80	1,90	2,77	3,85	3,31	3,19	4,06	3,94	3,89
K, min⁻¹	1,99	1,63	0,83	1,70	3,71	2,39	2,21	3,86	3,82	3,61
Δ weight, %	3,6	4,4	4,8	3,1	1,8	2,9	2,3	1,44	1,6	1,3
3, °C	220-300	230-300	218-299	220-290	200-250	220-270	200-260	190-250	190-230	200-230
R²	0,9926	0,9960	0,9989	0,9968	0,9890	0,9920	0,9902	0,9891	0,9993	0,9995
F_{cal}>F_{cr}	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
E_a, kJ/mol	66,43	74,69	71,80	64,46	69,03	90,65	76,04	75,33	88,89	84,98
n	7,99	8,99	8,64	7,75	8,31	10,91	9,15	9,06	10,70	10,226
K, min⁻¹	10,48	12,74	11,78	9,75	10,46	16,37	12,53	11,75	14,33	14,05
Δ weight, %	25,3	25,9	28,9	26,3	22,8	29,5	30,5	29,1	39,9	27,9
4, °C	310-420	310-430	300-518	300-520	260-300	270-310	260-300	260-310	240-330	240-320
R²	0,9905	0,9917	0,9777	0,9757	0,9990	0,9853	0,9988	0,9991	0,9853	0,9831
F_{cal}>F_{cr}	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
E_a, kJ/mol	32,04	31,09	24,05	23,03	75,85	82,77	87,97	77,73	44,79	38,30
n	3,86	3,74	2,89	2,77	9,13	9,96	10,59	9,35	5,39	4,61
K, min⁻¹	4,61	4,32	2,36	2,25	16,23	18,12	19,15	15,43	6,83	7,16
Δ weight, %	8,2	8,5	12,8	15,8	8,9	5,9	9,8	10,6	10,2	15,0
5, °C	430-510	440-530	see stage 4	see stage 4	300-460	310-490	310-480	310-510	330-500	330-500
R²	0,9701	0,9714			0,9470	0,9586	0,9727	0,9403	0,9302	0,9278
F_{cal}>F_{cr}	Yes	Yes			Yes	Yes	Yes	Yes	Yes	Yes
E_a, kJ/mol	72,07	72,70			37,15	33,48	31,34	29,52	33,43	32,78
n	8,67	8,75			4,47	4,03	3,77	3,55	4,02	3,94
K, min⁻¹	13,24	13,72			4,49	3,78	3,62	3,00	4,22	3,93
Δ weight, %	4,6	3,9	10,1	8,4	9,9	10,3	10,2	11,9		
6, °C	530-1080	530-890	528-859	530-1070	470-1100	490-1090	490-1090	520-1100	510-1050	510-1050
R²	0,9415	0,9604	0,9444	0,9462	0,9194	0,9228	0,9478	0,8948	0,8762	0,8641
F_{cal}>F_{cr}	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
E_a, kJ/mol	22,81	30,37	30,48	23,10	16,41	19,67	20,15	19,59	18,17	18,08

n	2,74	3,65	3,67	2,78	1,97	2,37	2,43	2,36	2,19	2,18
K, min⁻¹	1,57	3,25	3,13	1,77	0,79	1,16	1,10	1,15	1,08	1,13
Δ weight, %	1,9	1,3	1,2	1,6	3,0	2,2	4,2	1,9	1,7	1,9
7, °C	1090-1250	No	No	1080-1250	1140-1240	1100-1250	1100-1250	1140-1250	1070-1240	No
R²	0,9657			0,8220	0,9158	0,9397	0,9172	0,9027	0,9279	
F_{cal} > F_{cr}	Yes			Yes	Yes	Yes	Yes	Yes	Yes	
E_a, kJ/mol	200,8			167,86	224,02	227,79	145,08	172,12	117,32	
n	24,16			20,2	26,96	27,41	17,46	20,71	14,12	
K, min⁻¹	43,8	34,82	47,15	47,31	29,04	35,90	23,95			
Δ weight, %	0,2	0	0,2	0,1	0,2	0,2	0,5	0,1	0,1	0
Total weight loss, %	46,4	45,4	54,2	48,6	48,5	51,1	58,6	54,6	64,8	59,0

At the second stage of processing the results of thermolysis, the Freeman-Carroll differentiation method (4) [3] was used:

$$\frac{-(E/2,3R)\Delta(\frac{1}{T})}{\Delta \lg \Delta m_r} = -n + \frac{\Delta \lg(dm/dt)}{\Delta \lg \Delta m_r} \quad (4),$$

where $\Delta m_r = \Delta m_c - m$; Δm_c is the maximum mass loss; Δm is the total mass loss at time t .

Pretreatment of the change in the process speed and its differentiation in time made it possible to confirm stepwise destruction and to reveal the occurrence of several decomposition processes in certain temperature ranges (Fig. 5).

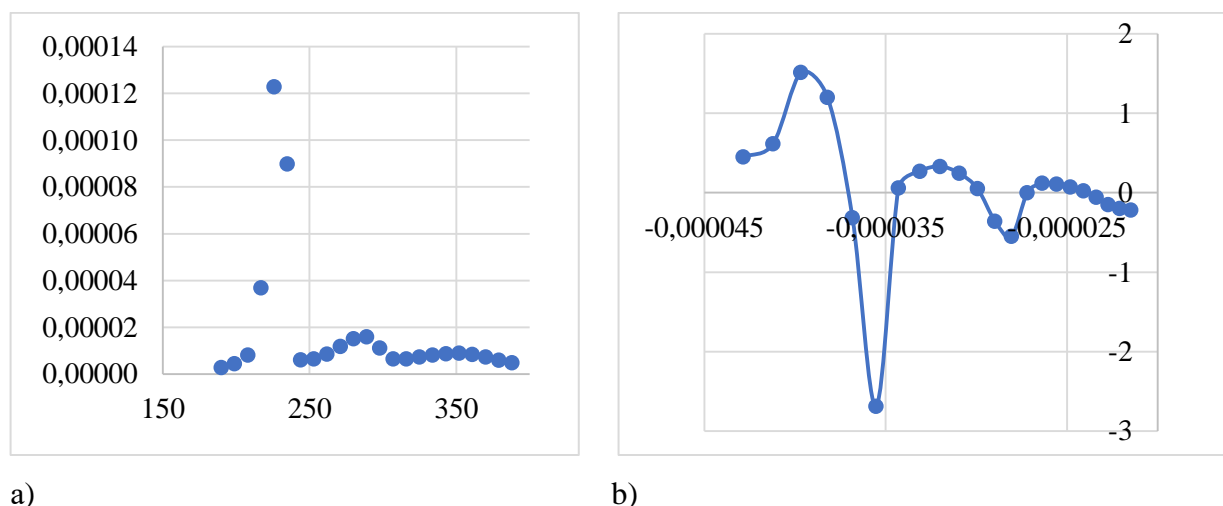


Figure 5. Dependences of the change in the process rate (a) and its time derivative (b) for the sample obtained in the presence of 20% IEDG in the range of 180-400⁰C.

TG/DTG curves were processed by temperature differentiation from the beginning to the end of the destruction process over the entire data array. The step of differentiation was 0,5 degrees (Fig. 6).

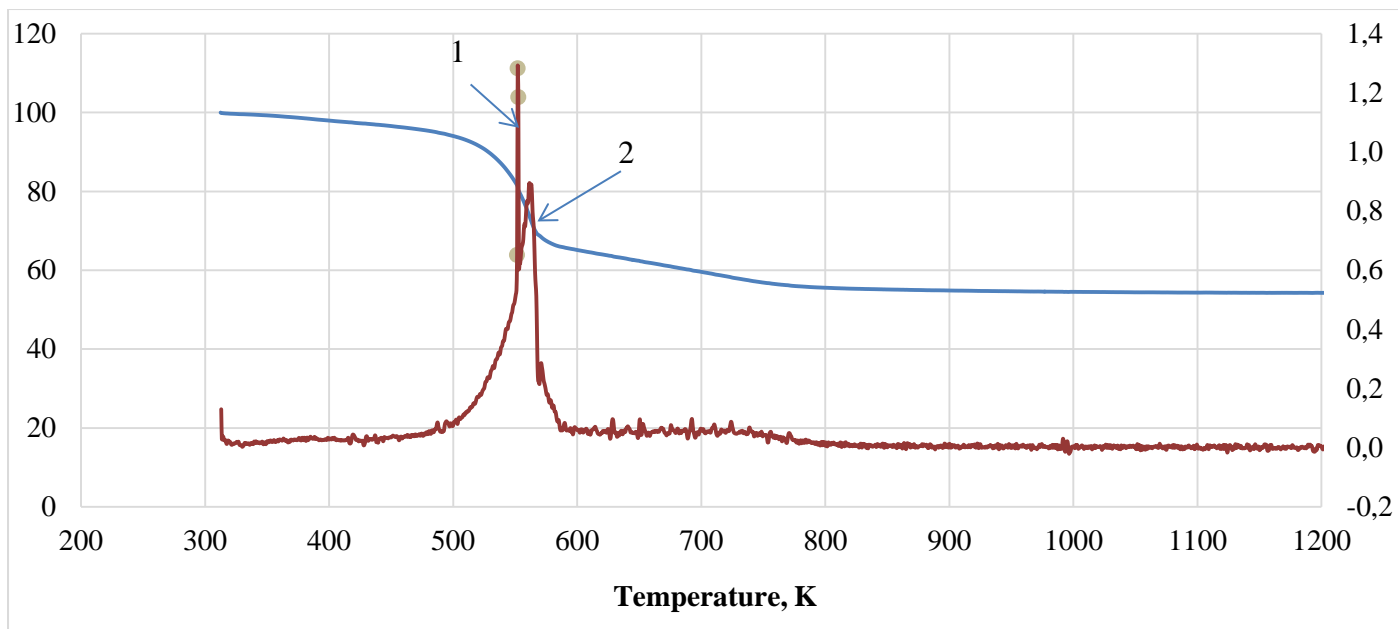


Figure 6. Differential curve of pure aluminum oxyhydrate.

The indicated method determined the characteristic peaks of mass loss from thermograms of all samples. For each selected area, the main characteristics of the process were calculated (Table 2).

Table 2 - Process characteristics of the main mass loss of a substance.

Main characteristics	Average values for the corresponding % IEDG				
	0	1	5	10	20
Temperature range, K	468-571	484-566	476-534	483-535	486-503
Peak Width, K	103	82	58	52	17
E_a , kJ/mol	34	73	84	87	180
Mass at the end of the decomposition section, %	68	65	69	66	62
Maximum speed, t^{-1}	0,8	0,7	2,3	3,0	8,2
Temperature for maximum speed, K	561	552	521	520	498
Width (peak 1), K	1,5	1,0	1,5	1,0	1,0
Temperature (peak 1), K	554	538	516	512	494
Δ mass (peak 1), %	2,0	1,9	2,2	1,9	2,0
Peak Speed 1, t^{-1}	1,3	1,3	1,4	1,3	1,6
Temperature range (peak 2), K	-	-	527-700*	535-577	504-570
E_a (peak 2), kJ/mol	-	-	16*	25	29
Mass at the end of the decomposition section (peak 2), %	-	-	56*	57	54
Maximum speed (peak 2), t^{-1}	-	-	0,2*	0,3	0,2
Temperature for maximum speed (peak 2), K	-	-	550*	555	557

*Allocated for one 5% sample.

To reveal the laws of thermal transformations of the samples, all differential curves were combined (Fig. 7).

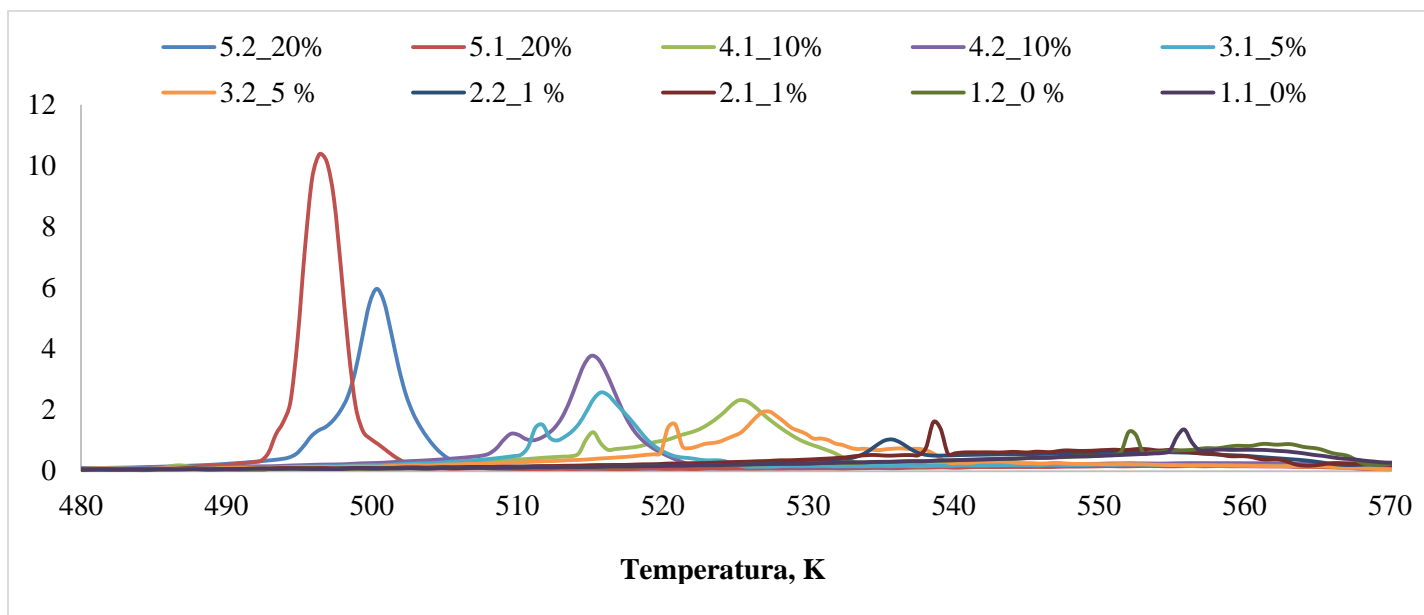


Figure 7. Combined temperature differentiation diagram for all samples.

The combined diagram made it possible to trace the shift in the temperature ranges characterizing the stages of the main mass loss of the substance depending on the IEDG content during the synthesis of the samples.

Discussion

The analysis of the experimental and calculated data and their interpretation was carried out from the standpoint of generally accepted ideas about the process of conversion of various forms of aluminum oxyhydrates into anhydrous aluminum oxide during thermolysis, and taking into account the synthesis conditions of the samples. According to the data of [8; 9; 10; 11; 12; 13], this process is associated with the existence of several metastable forms of alumina, transforming one into another with increasing temperature successively from $\text{Al}(\text{OH})_3$ to $\text{AlO}(\text{OH})$ and then to $\gamma\text{-Al}_2\text{O}_3$, $\delta\text{-Al}_2\text{O}_3$, $\theta\text{-Al}_2\text{O}_3$ и $\alpha\text{-Al}_2\text{O}_3$. The values of the transformation temperature between these forms depend on the purity of the precursors, their degree of crystallinity, and other factors [8].

The interval of heating the samples to 110-120⁰C (Table 1), apparently, is primarily associated with the removal of adsorbed water and alcohol residues, which is confirmed by the minimum values of all kinetic parameters. At temperatures of 70-90⁰C, dehydration of the residues of the initial $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and the initiation of its transition to $\text{Al}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ are possible [12]. At a temperature of 110⁰C,

decomposition of ammonium nitrate begins [15], which is one of the inevitable products in the synthesis of samples.

At the second distinguished stage of thermolysis (Table 1), with an increase in the IEDG content, a slight shift in the heating interval is observed from 120-220⁰C to 120-190⁰C. In our opinion, the upper limit of these temperature ranges $\geq 180^{\circ}\text{C}$ corresponds to the loss of the first portions of chemically bound water and the beginning of the formation of boehmite $\text{AlO}(\text{OH})$, which is consistent with the data [11; 12; 13]. This process is accompanied by thermal decomposition of aluminum nitrate residues, which begins at 150-170⁰C [14] with the release of NO_2 , O_2 , H_2O . This stage of thermal decomposition can correspond to the ongoing decomposition of ammonium nitrate, the mechanism of which proceeds in different directions and changes with temperature [15]. These processes are characterized by an increase in activation energy, parameter (n) and process speed, which correlate with the onset of product degradation. There were no significant differences in the nature of the thermal decomposition of aluminum oxyhydrate samples at the initial stages of thermolysis. However, the general pattern is to reduce the weight loss from 2,6 % to 1,0 % in the first stage and from 3,6% to 1,3% in the second stage of decomposition in the series “pure aluminum oxyhydrate - product with 20% IEDG”, bypassing sample with 1% surfactant content.

It was found that the temperature range corresponding to the main loss of substance $\sim 30\%$ shifts from 230-300⁰C for samples without IEDG to 220-230⁰C for samples synthesized in the presence of a surfactant. This stage proceeds with a significant increase in all kinetic parameters. Obviously, certain phase transitions of the product correspond to this process. Thermal decomposition during heating in air can occur in two directions: with the formation of boehmite and the amorphous alumina phase upon heating $\geq 250(300)^{\circ}\text{C}$ [11; 12; 13]. The presence of these transformations is confirmed by the calculated level of the apparent activation energy of 70–90 kJ/mol, as well as by the value of the parameter $n > 4$, which characterizes the increasing rate of formation of crystallization centers [4]. The estimated mass loss of such a conversion of the corresponding dehydration during the destruction of boehmite is 15,0 wt.% [8]. An additional loss of substance is most likely observed due to the decomposition of residues of aluminum nitrate, ammonium nitrate and in connection with the start of IEDG decomposition. The characteristics of the process of the main mass loss of a substance, determined by Freeman-Carroll differentiation (Table 2), naturally change with increasing amount of surfactant. In the series “pure aluminum oxyhydrate - a product with 20% IEDG”, the peak width decreases from 103 to 17K, and the value of the apparent activation energy increases from 34 to 180 kJ/mol. The maximum destruction rate increases from 0,8 to 8,2 min^{-1} . The temperature of the maximum speed of the process shifts from 561K to 498K, which is consistent with the calculated values obtained by the procedure [1] (Table 1).

It should be noted that in the range 230-290⁰C for all samples, in addition to the main destruction, a section with fast and intense mass loss in a narrow temperature range with the characteristics indicated in (Table 2) is distinguished. The peak width of this section with conditional number 1 (Fig. 6, Table 2) is 1,0-1,5K, and the mass loss is $\sim 2\%$. The peak temperature varies from 494K for the sample with 20% IEDG to

554K for pure aluminum oxyhydrate. Such a process can be explained by the final decomposition of ammonium nitrate, which can proceed especially rapidly in a given temperature range [15].

With further heating from 300 to 500⁰C, destruction occurs in different ways depending on the content of IEDG. For samples obtained without surfactants, this interval is divided into steps 310-420⁰C and 430-530⁰C, which correspond to crystallization of alumina that occurs in two directions of phase transitions [11; 13]. In samples with 1% IEDG, this interval is combined. For products with 5–20% IEDG, the maxima of the kinetic parameters remain no higher than 300⁰C, and stabilize at approximately the same level with increasing temperature (Table 1). It should be noted that weight loss at a temperature of 350-400⁰C can be associated with the burnout of IEDG [6].

When heated from 500 to 1000⁰C, the kinetics of the process is described by close characteristics and involves the formation and crystallization of metastable forms of aluminum oxides. At the last isolated stage of thermolysis for seven out of ten samples, there is a sharp increase in all kinetic parameters with a slight mass loss of the order of 0,2% (Table 1). The average value of the apparent activation energy of 180 kJ/mol is associated with the formation of corundum (α -Al₂O₃) [2]. The formation of a new phase and the course of crystallochemical processes is also confirmed by the high values of parameter (n) and the rate of the process.

It was noted that the total mass loss in the series of samples 0, 1, 5, 10, 20% of IEDG naturally increases in proportion to the surfactant content.

Thus, based on the thermal analysis data, the kinetic parameters of the thermal decomposition of aluminum oxyhydrates obtained by the hydrolysis of aluminum nitrate in the presence of IEDG and without the use of a surfactant were calculated and analyzed. The characteristic temperature range of the main mass loss of the substance is established. The sequence of transformations that occur during thermal destruction of the samples is determined. The patterns of changes in the kinetics of transformations under the influence of different concentrations of surfactants are described.

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