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INFLUENCE OF LIME-CONTAINING ADDITIVES ON THE THERMAL BEHAVIOUR OF AMMONIUM NITRATE

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Ammonium nitrate (AN) is one of the main nitrogen fertilizers used in fertilization programs. However, AN has some serious disadvantages – being well soluble in water hardly 50% of the N-species contained are assimilated by plants. The second disadvantage of AN is associated with its explosive properties. The aim of this paper was to clarify the influence of different lime-containing substances – mainly Estonian limestone and dolomite – as internal additives on thermal behaviour of AN.

Commercial fertilizer grade AN was under investigation. The amount of additives used was 5, 10 or 20 mass%, or calculated on the mole ratio of AN/(CaO, MgO)=2:1 in the blends. Experiments were carried out under dynamic heating condition up to 900°C (10°C min^{-1}) in a stream of dry air or N_2 by using Setaram Labsys 2000 equipment coupled to Fourier transform infrared spectrometer (FTIR).

The results of analyses of the gaseous compounds evolved at thermal treatment of neat AN indicated some differences in the decomposition of AN in air or in N_2. At the thermal treatment of AN's blends with $CaCO_3$, $MgCO_3$, limestone and dolomite samples the decomposition of AN proceeds through a completely different mechanism – depending on the origin and the content of additives, partially or completely, through the formation of $Mg(NO_3)_2$ and $Ca(NO_3)_2$.

Keywords: *ammonium nitrate, dolomite, limestone, TG-FTIR, thermal stability*

Introduction

Ammonium nitrate (AN) is one of the main nitrogen (N) mineral fertilizers the consumption of which in 2005 was on the level of 13 million tons [1]. AN is characterized by high content of nitrogen (35%) and relatively simple manufacturing technology. However, AN has two serious disadvantages – being well soluble in water hardly 50% of N-species contained are assimilated by plants [2–4]. The other part of N is lost by leaching of N-species [3–5] or emission of them into the atmosphere [3–7]. The second disadvantage of AN is associated with its explosive properties – AN is a light explosive with upset oxygen balance which, however, can be easily restored by adding hydrocarbon components of any origin.

There are two widespread possibilities to reduce above mentioned disadvantages of AN – internal additives or coating agents can be used for modification of AN to obtain the controlled-realized fertilizers (CRF) [2, 8–11].

The thermal behavior and the possibilities of enhancing the thermal stability of AN have been studied by several authors [12–20]. AN has six temperature dependent solid-state phases at normal pressure. The principal transitions are $AN_{VI}\leftrightarrow AN_{V}\leftrightarrow AN_{IV}\leftrightarrow AN_{III}\leftrightarrow AN_{II}\leftrightarrow AN_{I}\leftrightarrow AN_{melt}$ with equilibrium temperatures T_e –170, –17, 32...55, 84, 125 and 170°C, respectively. In addition, a metastable transition $AN_{IV}\leftrightarrow AN_{II}$ is known at T_e 50°C [14]. The phase transition $AN_{IV}\leftrightarrow AN_{III}$ associated with a large volume change of the AN crystal causes cracking of the particles with the following decrease in the thermal stability of AN. From technological point of view at manufacturing or at handling of AN the phase transition $AN_{IV}\leftrightarrow AN_{II}$ should be followed, by-passing the form AN_{III}.

In [12] the influence of reactive-grade sodium, potassium, ammonium, calcium and magnesium sulphate, phosphate and carbonate additives on the thermal stability of AN were studied. Most of these additives shifted the maximum of decomposition exotherm of AN towards higher temperatures. The best stabilizing effect on AN was obtained with carbonate additives and at that calcium carbonate stabilized AN better than sodium, potassium or magnesium carbonate.

Data on the influence of natural and technical mineral additives on the thermal stability of AN is almost missing. Considering the positive influence of reactive-grade Ca- and Mg-carbonates on the thermal characteristics of AN, natural limestone and dolomite which contain in addition to the main compounds $CaCO_3$ and $CaCO_3\cdot MgCO_3$ also different impurities, would be suitable minerals for improving thermal stability of AN.

The aim of this paper was to study the effect of different lime-containing substances as internal additives on AN thermal stability, mainly concentrating on Estonian limestone and dolomite as potentially suitable coating agents for AN granules.

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Experimental

Materials

Commercial granulated fertilizer-grade AN (34.4% N) (Tserepovetski Azot Ltd., Russia) was under investigation. Three Estonian limestone (from Võhmuta, Vasalemma and Karinu deposits) and two dolomite (Kurevere and Anelema) samples previously ground (<45 μm) were used as additives to AN. Magnesium and calcium carbonates used were of analytical-grade (VWR International Ltd.). The chemical analyses presented in Table 1 show that all samples are relatively pure limestone and dolomite. The content of total CaO in limestone was between 52.9–54.2 mass% and of total MgO 1.1–2.8 mass%. In dolomite samples the content of total CaO was on the level of 29.0 mass% and of total MgO 24.4–26.6 mass%.

The SSA of the studied samples, given in Table 1 was relatively small (from 0.74 to 2.44 $m^2 g^{-1}$), being somewhat bigger for dolomite samples. Hereby, the selected samples were slightly porous and well crystallized.

The quantitative XRD study confirmed the pure and well crystallized nature of the samples, especially, for the limestone ones. In limestone the content of calcite [$CaCO_3$] was between 93.4% (Karinu) and 97.1% (Vasalemma) and the content of dolomite [$CaMg(CO_3)_2$] between 2.2% (Vasalemma) and 6.3% (Karinu). The content of [$CaMg(CO_3)_2$] in Kurevere and Anelema samples was 96.4 and 92.6%, respectively. The other minerals like quartz [SiO_2], illite–smectite [$Na,K_x(Al,Mg)_2Si_4O_{10}(OH)_2xH_2O$], K-feldspar [$KAlSi_3O_8$], albite and pyrite were present on the level of 0.3–1.7% or as traces.

Methods

To study the influence of additives on the thermal stability of AN, thermogravimetric equipment (Setaram Labsys 2000) capable of simultaneous recording of mass loss (TG), differential mass loss (DTG) and differential thermal analyses curve (DTA) was used. The experiments were carried out under dynamic heating conditions from 30 to 900°C, or in the case of neat AN to 400°C, at a heating rate of 10°C min^{-1} in a stream of dry air or N_2 (flow rate 120 mL min^{-1}). Standard 100 μL alumina crucibles were used. Sample mass was calculated on the basis of constant mass of AN (15±0.5 mg), the content of additives in the blends being 5, 10 or 20 mass%, or calculated from the mole ratio of AN/(CaO+MgO)=2:1.

To identify the gaseous compounds emitted during the thermal treatment of samples, Interspec 2020 Fourier Transform Infrared Spectrometer (FTIR) was used. The measurements were recorded in the 4000–600 cm^{-1} region with the resolution of 4 cm^{-1} and an average out of four scans was taken. To identify the gaseous compounds, the Bio-Rad (Sadtler) KnowItAll search program and Gases & Vapours Database (code GS) were used.

Results and discussion

Thermal analysis

From the DTA curves of ground granular AN samples four endotherms between 30 and 180°C indicating to phase changes could be observed (Fig. 1). The first, $AN_{IV}\leftrightarrow AN_{III}$ transition with minimums on DTA curve in air and in nitrogen atmosphere was fixed at 53.0 and 53.7°C, respectively. The $AN_{III}\leftrightarrow AN_{II}$ and $AN_{II}\leftrightarrow AN_{I}$ transitions followed with minimums at 88.1 and 90.8°C, and at 127.1 and 128.0°C. The $AN_{I}\leftrightarrow AN_{melt}$ transition endotherms had its minimums at 164.8 in air and 168.4°C in nitrogen. Further increasing of temperature reveals a massive exothermic effect characterizing the decomposition of AN. The decomposition exotherm had its maximums at 263.0°C (with a shoulder at 246.4°C) in air and a double peak with maximums at 276.0 and 286.3°C (and with a shoulder at 269.1°C) in nitrogen. So, there was observed not high but systematic increase (0.7–2.7°C) in transition temperatures of AN in nitrogen atmosphere as compared to these obtained in air, whereas the exothermic maximum of AN decomposition in nitrogen was moved 13°C towards higher temperature and, ac-

Table 1 Chemical composition and specific surface area (SSA) of limestone and dolomite samples

Sample	Chemical composition/mass%[*]							BET SSA/$m^2 g^{-1}$
	CaO	MgO	CO_2	I.R.[**]	Al_2O_3	Fe_2O_3	$S_{sulfate}$	
Karinu	52.92	2.76	38.98	0.80	1.72	0.08	0.04	0.74
Võhmuta	53.17	1.50	39.87	1.33	1.81	0.04	0.06	1.56
Vasalemma	54.22	1.14	40.45	0.75	1.50	0.09	0.09	0.89
Kurevere	29.04	24.40	41.87	3.17	0.64	0.21	0.09	1.70
Anelema	28.85	26.63	40.81	5.83	0.84	0.14	0.10	2.44

[*]Per dry mass, [**]Insoluble residue in aqua regia

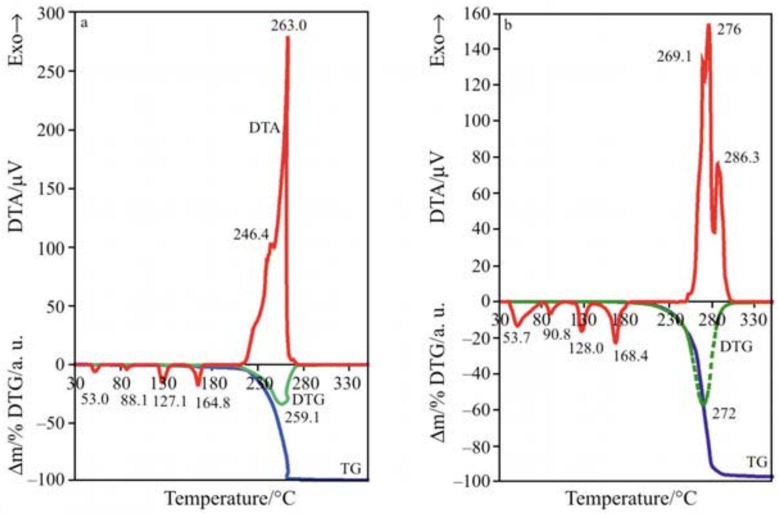

Fig. 1 Thermal curves of AN in a – air and b – nitrogen

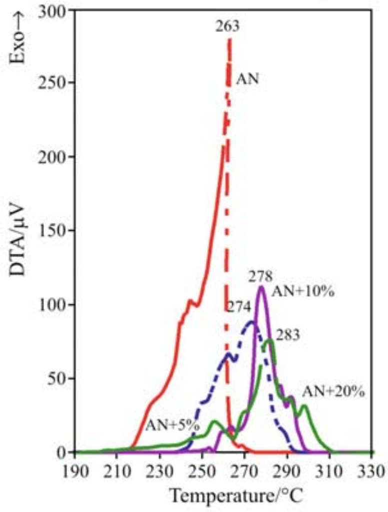

Fig. 2 DTA curves of AN and its blends with Kurevere dolomite

cording to the shape of the DTA curve, the decomposition had a multi-step character. The total mass loss up to 315°C was, for both, 97–98%.

Limestone–dolomite additives in the amount of 5–10 mass% did not affect the $AN_{IV}\leftrightarrow AN_{III}$, $AN_{III}\leftrightarrow AN_{II}$ and $AN_{II}\leftrightarrow AN_{I}$ phase changes in a systematic manner, but the melting point and the decomposition temperature of AN were shifted (except with Vasalemma limestone), respectively, 1.5–1.7 and 8–15°C towards higher temperatures (Table 2, Fig. 2). Vasalemma limestone in the amount of 5 mass% moved the temperature of $AN_{II}\leftrightarrow AN_{I}$ phase transition and the melting point a few degrees towards lower, but the maximum of exotherm 45.8°C towards higher temperature – to 308.8°C. Vasalemma limestone was characterized as the purest and most well crystallized sample studied (Table 1).

If the amount of additives was 20 mass% or more, the $AN_{IV}\leftrightarrow AN_{II}$ phase transition instead $AN_{IV}\leftrightarrow AN_{III}$, $AN_{III}\leftrightarrow AN_{II}$ was observed on DTA curve, and 20 mass% of Kurevere dolomite shifted the exothermic maximum 20°C towards higher temperature (Fig. 2). The total mass loss of AN blends with 5, 10 and 20% Kurevere dolomite up to 315°C was 80, 71 and 56% from the initial sample mass, respectively.

On the DTA curves of AN blends with limestone or dolomite samples (as well as with reactive-grade $CaCO_3$ or $MgCO_3$) at the mole ratio of AN/(CaO+MgO)=2:1 the decomposition exotherm of AN was disappeared and a multi-peaked endotherm was fixed on the DTA curves in the temperature interval of 200–300°C (Fig. 3), characterizing the interaction of AN with $CaCO_3$ and $MgCO_3\cdot CaCO_3$, contained in nat-

Table 2 Thermal characteristics of AN with different limestone and dolomite samples (5 mass%) in air

Compound	Phase change/°C				Exotherm maximum/°C
	$AN_{IV}\leftrightarrow AN_{III}$	$AN_{III}\leftrightarrow AN_{II}$	$AN_{II}\leftrightarrow AN_{I}$	$AN_{I}\leftrightarrow AN_{melt}$	
AN	53.6	88.1	127.1	164.8	263.0
AN+Võhmuta	53.0	88.4	127.0	166.5	274.3
AN+Karinu	52.5	90.9	127.8	166.3	274.3
AN+Vasalemma	53.0	88.1	125.8	161.4	308.8
AN+Kurevere	52.5	87.6	127.8	166.4	273.8
AN+Anelema	50.7	89.2	126.2	166.3	270.7

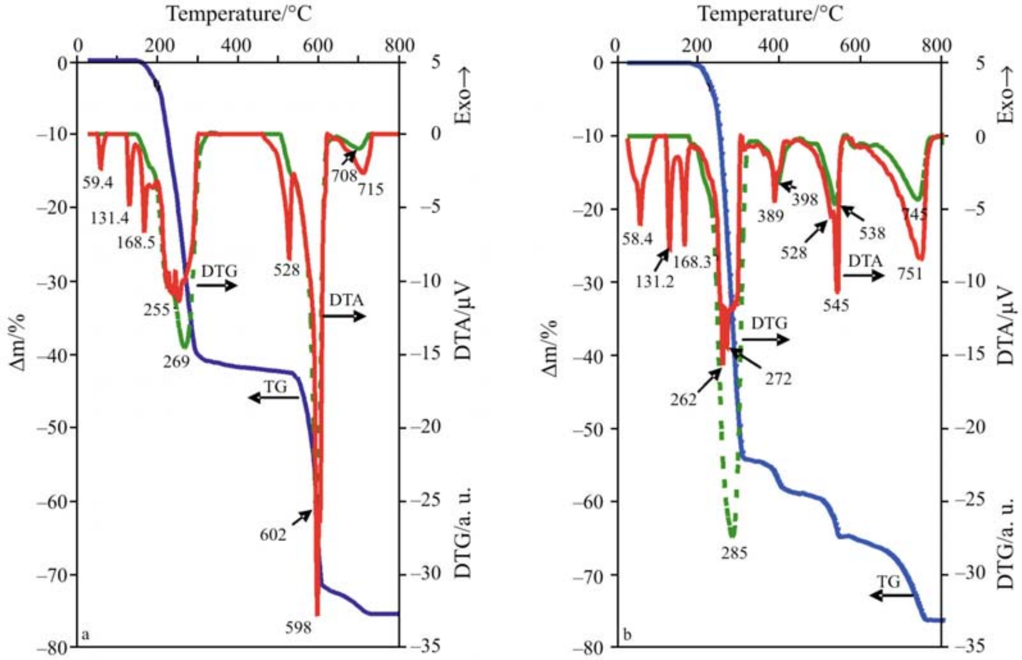

Fig. 3 Thermal curves of AN blends with a – Karinu limestone and b – Kurevere dolomite at mole ratio of AN/(CaO+MgO)=2:1 in air

ural limestone and dolomite samples, with the formation of calcium and magnesium nitrates. The total mass loss at heating up to 315°C was 54.1% for Kurevere dolomite and 40.7% for Karinu limestone.

The followed endotherms with minimum on DTA curves at 389°C (Fig. 3b) and double-peak at 528 and 545°C (Fig. 3b) and at 528 and 598°C (Fig. 3a) characterized the decomposition of previously formed $Mg(NO_3)_2$ and $Ca(NO_3)_2$, respectively [21, 22]. The last endotherms on DTA curves with minimums at 751 (Fig. 3b) and 715°C (Fig. 3a) describe the decomposition of residual carbonates. The total mass loss at heating of the samples up to 800°C for Kurevere dolomite and Karinu limestone was 76.2 and 75.3%, respectively. For blends with lower content of limestone and dolomite more modest decomposition endoeffects of nitrates and carbonates were fixed at the same temperature intervals.

FTIR analysis

The results of the analysis of gaseous compounds evolved at the thermal treatment of neat AN indicated to some differences in the decomposition of AN carried out in air or in N_2. In air the main products of AN decomposition were N_2O (2235 and 2215 cm^{-1}) and H_2O (bands in the broad ranges 3900–3500 and 1900–1300 cm^{-1}) as the literature suggests [12, 15], but also the peaks characteristic to NO_2 (1630 and 1595 cm^{-1}) and to NH_3 as traces (967 and 392 cm^{-1}) could be fixed on the FTIR spectra (Fig. 4a). The peaks at 1630 and 1595 cm^{-1} were partially overlapping water peaks in the same region. The emission of N_2O and NO_2 at 220–320°C was the most intensive at 280°C. These temperatures correspond well to the data received from thermal analysis (Fig. 1a). The emission of the same compounds in the temperature range of 230–320°C as in air, can be fixed also on the FTIR spectra of neat AN in N_2, but in addition, clear and intensive NH_3 peaks can be detected in the 1200–750 cm^{-1} region (Fig. 4b). This may be due to the fact that the first step of AN's decomposition is dissociation into NH_3 and HNO_3 [12, 15]:

$$NH_4NO_3 \rightarrow NH_3 + HNO_3 \tag{1}$$

and N_2 is suggested to be one by-product of AN's decomposition.

$$5NH_3 + 3HNO_3 \rightarrow 4N_2 + 9H_2O \tag{2}$$

The presence of it in larger quantities inhibit the further decomposition of AN into N_2O and H_2O by the summary equation:

$$NH_4NO_3 \rightarrow N_2O + 2H_2O \tag{3}$$

The results of the analysis of gaseous compounds evolved at the thermal treatment of AN and $CaCO_3$ or Karinu limestone blends at the mole ratio of AN/(CaO+MgO)=2:1 suggested that when mixed with $CaCO_3$ or Karinu limestone, the decomposition of AN proceeds through a completely different mechanism. None of the peaks indicating to the emission of N_2O could be fixed on the FTIR spectra of the AN blend with $CaCO_3$ in N_2, and the maximum intensities of the characteristic peaks of N_2O in air as well as for

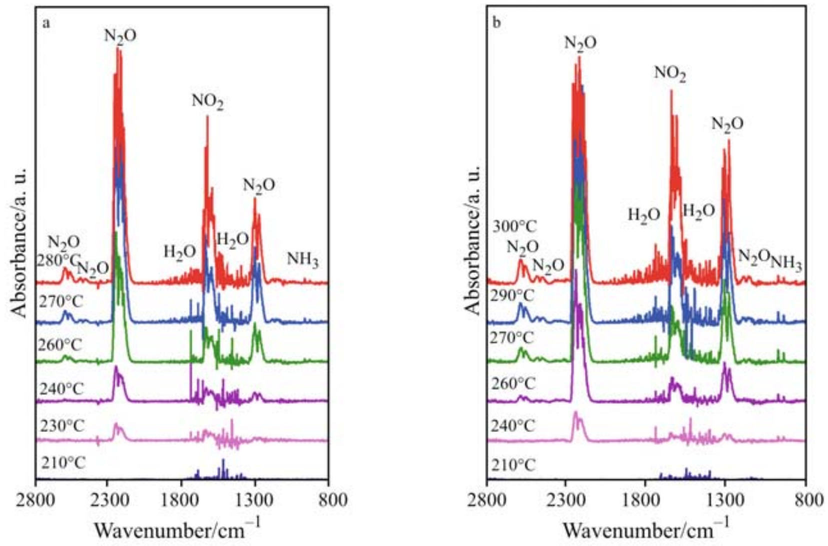

Fig. 4 FTIR spectra of gaseous compounds evolved at thermal treatment of AN in a – air and b – nitrogen

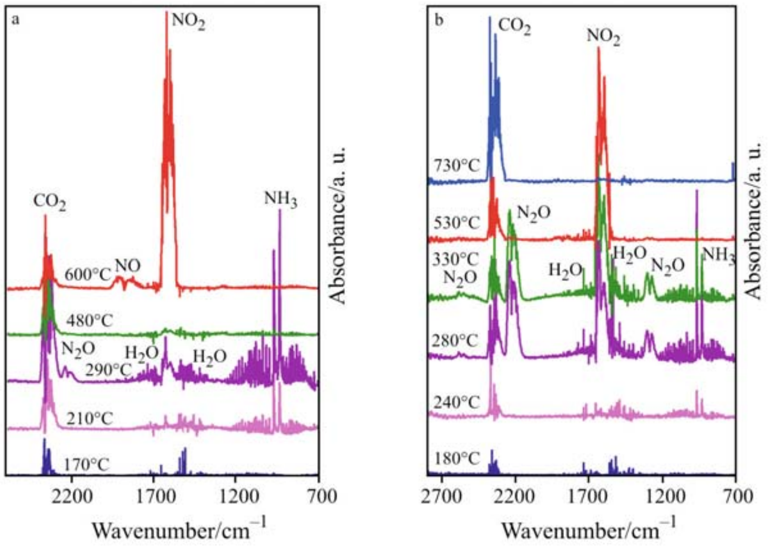

Fig. 5 FTIR spectra of gaseous compounds evolved at thermal treatment of AN blends with a – Karinu limestone and b – Kurevere dolomite at mole ratio of AN/(CaO+MgO)=2:1 in air

AN blend with Karinu limestone both in air (Fig. 5a) and in nitrogen, were relatively low (Fig. 6).

In air in the temperature region from 180 to 300°C and from 200 to 320°C for AN blends with $CaCO_3$ and Karinu limestone, respectively, the peaks characteristic to NH_3 appeared in the 1200–750 cm^{-1} region. CO_2 (2367 and 2354 cm^{-1}) emitted most intensively in the temperature range between 240 and 300°C, but traces of it can be detected on the FTIR spectra up to 800°C due to the decomposition of residual carbonates.

In air modestly intensive characteristic peaks for NO_2 were fixed on the FTIR spectra in the temperature interval from 200 to 290°C, however, it starts to emit more intensively at 500°C – at the temperature when precedingly formed $Ca(NO_3)_2$ (Eq. (4)) begins to decompose having at that with the maximum peak intensities for the blends with $CaCO_3$ and Karinu limestone at 580 and 610°C, respectively.

$$2NH_4NO_3+CaCO_3 \rightarrow Ca(NO_3)_2+2NH_3+CO_2+H_2O \quad (4)$$

In the temperature interval from 500 to 630°C emission of a compound giving peaks in the range of 1800–1950 cm^{-1} can be detected which is most likely characteristic to NO [23] evolved during decomposition of $Ca(NO_3)_2$.

The formed $Ca(NO_3)_2$ decomposes at temperatures over 500°C. In [22] various decomposition reactions for $Ca(NO_3)_2$ are suggested, of which probably Eqs (5) and (6) are dominating and Eq. (7) occurs

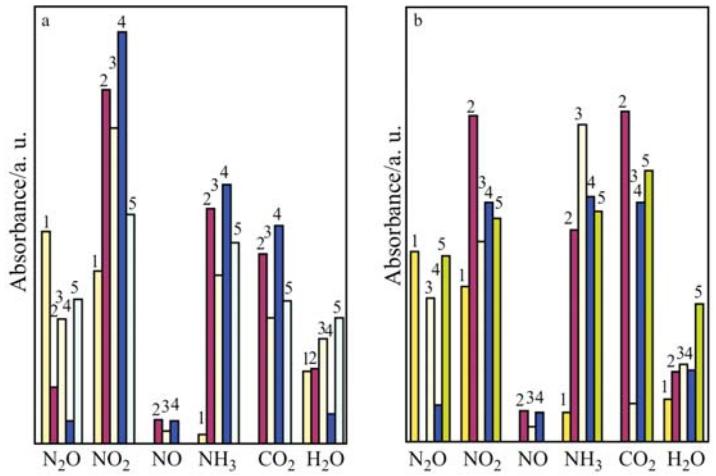

Fig. 6 Maximum intensities of characteristic peaks of gaseous compounds evolved at thermal treatment of AN and its blends with different lime-containing additives at mole ratio of AN/(CaO+MgO)=2:1 in a – air and b – nitrogen; 1 – AN, 2 – AN+$CaCO_3$, 3 – AN+$MgCO_3$, 4 – AN+Karinu, 5 – AN+Kurevere

as a side reaction under the given experiment conditions, because on the FTIR spectra the characteristic peaks of NO_2 are the most intensive and the ones of NO somewhat less:

$$Ca(NO_3)_2 \rightarrow CaO+NO_2+NO+O_2 \quad (5)$$

$$Ca(NO_3)_2 \rightarrow CaO+2NO_2+1/2O_2 \quad (6)$$

$$Ca(NO_3)_2 \rightarrow CaO+2NO+11/2O_2 \quad (7)$$

The results of the FTIR analysis of gaseous compounds like CO_2, NO_2, NO evolved at the thermal treatment of the same blends in N_2 were similar to the ones in air, with the exception that the beginning and the maximum intensities of characteristic peaks were shifted 10–40°C towards higher temperatures (Table 3).

So, FTIR analysis suggests that AN mixed with $CaCO_3$ or limestone at the equivalent mole ratio reacts almost completely with $CaCO_3$ as in the temperature region of AN decomposition, in addition to NH_3 and CO_2, only modest amount of N_2O – the main product of AN decomposition – was evolved and the decomposition mechanism of AN was not heavily affected by the atmosphere used in the experiment.

The results of the FTIR analysis of gaseous compounds evolved at the thermal treatment of AN blends with $MgCO_3$ and Kurevere dolomite (Fig. 5b) at mole ratio 2:1 differed from the ones of blends with $CaCO_3$ and Karinu limestone (Fig. 6). At 250–270°C the main product of AN decomposition N_2O began to evolve, having the most intensive peaks on the FTIR spectra at 320–330°C both in air and in N_2. The intensities of the respective peaks for AN blends with $MgCO_3$ and Kurevere dolomite were much higher than obtained with $CaCO_3$ and Karinu limestone additives (Fig. 6). This indicates to the fact that the reaction of AN with $MgCO_3$ and dolomite was not as complete as with $CaCO_3$ and limestone, because $MgCO_3$ is less reactive towards AN than $CaCO_3$, and part of AN decomposed completely without reacting with $MgCO_3$. Also NH_3 and CO_2 emitting most inten-

Table 3 Temperatures (°C) of maximum intensities of characteristic absorption peaks of gaseous compounds evolved at thermal treatment of AN and its blends with lime-containing additives at mole ratio of AN/(CaO+MgO)=2:1

Sample	N_2O (2230 cm^{-1})	NO_2 (1630 cm^{-1})	NO (1845 cm^{-1})	NH_3 (967 cm^{-1})	CO_2 (2367 cm^{-1})	H_2O (1735 cm^{-1})
In air						
AN	280	300	–	280	–	230
AN+$CaCO_3$	330	580	580	280	240	280
AN+$MgCO_3$	330	480	480	260	260	330
AN+Karinu	280	610	620	280	280	230
AN+Kurevere	330	550	–	280	730	330
In nitrogen						
AN	290	300	–	270	–	300
AN+$CaCO_3$	–	630	630	280	280	260
AN+$MgCO_3$	320	480	480	260	330	280
AN+Karinu	330	680	680	280	280	330
AN+Kurevere	330	550	–	280	730	330

sively from 180 to 300°C and NO_2 from 350 to 480°C for the blend with $MgCO_3$, and from 350 to 550°C for the blend with Kurevere dolomite, indicate that AN reacts with $MgCO_3$ to a limited extent according to

$$2NH_4NO_3+MgCO_3\rightarrow Mg(NO_3)_2+2NH_3+CO_2+H_2O \qquad (8)$$

with the following decomposition of $Mg(NO_3)_2$ at higher temperatures.

Emission of NO was fixed only when using $MgCO_3$, but not Kurevere dolomite (Fig. 6). The peaks characteristic to CO_2 could be observed on the FTIR spectra of AN blend with Kurevere dolomite being most intensive at 730°C (Table 3) indicating to the decomposition of unreacted carbonates.

So, the proof of AN reaction with carbonates in dolomite was even more solid and obvious than in the case of limestone. The FTIR analysis of the gaseous compounds evolved at the thermal treatment of AN with 10–20 mass% of limestone–dolomite additives showed analogues, but less expressed tendencies.

Conclusions

There was no considerable systematic dependence of the temperatures of the $AN_{VI}\leftrightarrow AN_V\leftrightarrow AN_{IV}\leftrightarrow AN_{III}\leftrightarrow AN_{II}\leftrightarrow AN_I$ phase transitions on the 5–20 mass% of limestone or dolomite additives, but depending on the type and amount of additive, the melting point and the decomposition temperature of AN were shifted 1.5–2 and 8–45°C towards higher temperatures. If the amount of additives was 20 mass% or more, the $AN_{IV}\leftrightarrow AN_{II}$ phase transition instead of $AN_{IV}\leftrightarrow AN_{III}\leftrightarrow AN_{II}$ occurred, and at mole ratio of AN/(CaO+MgO)=2:1 an endoeffect instead of exoeffect was fixed on DTA curve. All the samples with lime-containing additives exhibited the endotherms on the DTA curves in the temperature range of decomposition of $Ca(NO_3)_2$ or $Mg(NO_3)_2$, suggesting the possibility of their formation during decomposition of AN.

The results of analysis of the gaseous compounds evolved during thermal treatment of neat AN indicated to some differences in the decomposition of AN in air and N_2. The thermal treatment experiments of AN blends with $CaCO_3$, $MgCO_3$, limestone and dolomite samples suggest that with blends the decomposition of AN proceeds through a completely different mechanism – depending on the origin and the content of additives, partially, or completely, through the formation of $Mg(NO_3)_2$ and $Ca(NO_3)_2$ which decompose at 370–630°C. The AN reaction with $MgCO_3$, and dolomite samples was not as complete as with $CaCO_3$ or limestone samples, because $MgCO_3$ is thermodynamically less reactive towards AN than $CaCO_3$.

Acknowledgements

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