

Thermal Stability of Ammonium Nitrate in Two-Component Mixtures with Powdered and Fine-Grained Materials

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Abstract. Ammonium nitrate(V) (AN, NH_4NO_3) is widely and widely used in the chemical industry, in agriculture as a fertilizer, explosive for military and civil purposes (e.g. in mining) or as a solid propellant [1, 2]. Storage of ammonium nitrate poses many problems, as it may be hazardous. This was proven, for example, by the explosion in 2020 in Beirut. Ammonium nitrate was stored in a warehouse at the port among other wares and an unfortunate turn of events caused a huge explosion. The explosion contributed to the formation of a 140-meter crater and an earthquake with a magnitude of 3.3 on the Richter scale. This explosion was classified as the third most destructive urban explosion of all time, after the atomic bombs in Hiroshima and Nagasaki at the end of World War II [3, 4], as the mixtures of oils (fuel or gas) and a concentrated form of nitrogen fertilizer – ammonium nitrate form explosives [5]. The dangerous properties of AN have been extensively studied. It is known that pure AN is stable at room temperature but may explode when mixed with impurities in a confined space or under fire-hazard conditions [1]. The research aimed to analyze the changes occurring in two-component mixtures with ammonium nitrate and powdered or fine-grained materials and to assess the effect of such an admixture on the fertilizer. Thermal analysis was used to carry out the TG-DSC tests.

Introduction

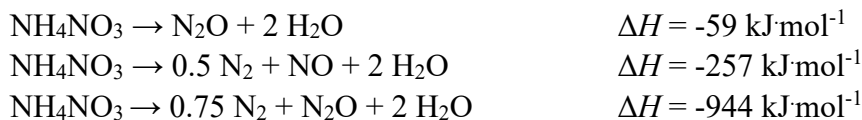
Ammonium nitrate(V) is a colorless, crystalline salt, well soluble in water. AN is hygroscopic and does not form hydrates. In explosives, the nitrate ion from ammonium nitrate is a source of oxygen and is used as an oxidant [6]. The most commonly used AN-based explosive is ANFO (Ammonium Nitrate Fuel Oil) due to its easy and inexpensive manufacturing. ANFO is produced by mixing ammonium nitrate with fuel oil (combustible component), typically in a weight ratio of 94:6. There are also other AN-based explosives, e.g. dynamon K (mixture with easily oxidized combustible substances, e.g.: 90% AN, 10% wood flour, ANNM (Ammonium Nitrate – NitroMethane) and ammonals (explosive mixtures consisting of TNT, AN and aluminum powder) [7-9].

The storage of ammonium nitrate for agricultural purposes is primarily connected with the problem of caking of such material or the hygroscopic nature of this compound. The clumping of AN is the result of adhesive forces and the process itself causes too much pressure on the layers located in the areas located lower during storage. Pressure contributes to changes in the physicochemical properties of AN. The chemical reactivity of AN has been well documented throughout the last century. Ammonium nitrate(V) can explode between 260°C and 300°C [6]. Interestingly, AN fertilizer, being non-flammable by nature, does not have explosive properties, unlike coal, wood, grain-based or other organic powders [10]. At sufficiently high temperatures, ammonium nitrate may decompose rapidly on its own. As a result, gases are formed, including nitrogen oxides and water **vapor**, and the rapid release of these gases causes an explosion [6, 10].

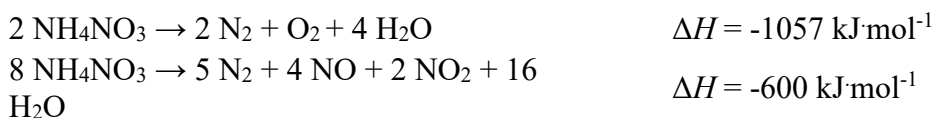
Different mechanisms of AN degradation have been described in the literature [6, 10-12], and the most accepted reactions are summarized below. The reversible reaction can occur at relatively low temperatures (i.e., ca 170°C). It is believed that the evaporation of molten AN leads to the formation of ammonia and nitric acid, which can initiate the decomposition of AN by the following reaction:



At higher temperatures (between 170°C and 280°C) irreversible exothermic reactions occur:



If the material is suddenly heated, explosive decomposition will occur [6, 10-12].



Ammonium nitrate(V) under standard pressure exists in five stable polymorphic forms (designated as phases I, II, II, IV and V; see Table 1).

Table 1. Crystallographic forms of ammonium nitrate [12]

Crystalline Phase	Temperature Range	Crystal System
I	169.5°C ... 125.2°C	Cubic
II	125.2°C ... 84.2°C	Tetragonal
III	84.2°C ... 32.3°C	Orthorhombic
IV	32.3°C ... -18°C	Orthorhombic
V	-18°C ... -103°C	Orthorhombic

Various types of admixtures for mixtures with AN can act in three ways: as an inhibitor, promoter or neutral additive. When the decomposition onset temperature is higher than that of pure AN, the additive is an inhibitor. When the decomposition onset temperature is lower than that for pure AN, the additive is considered a promoter. There is still a third option, the addition does not change the "initial" temperature of decomposition. The additives behave as inert materials that only thin the AN [12]. Additives in the form of inhibitors can mitigate the risk of AN explosion. As the literature data show, such properties showed:

- sodium, potassium, ammonium and calcium salts;
 - sulphates, phosphates, carbonates, organic substances (urea, oxalate, methanoate, guanidine salts);
 - sodium salts of weak acids (carbonic acid, acetic acid, formic acid, oxalic acid).
- On the other hand, chemical additives called promoters had completely different properties, they accelerate the potential explosion of AN. These include:
- nitrocellulose, aromatic nitro compounds;
 - non-explosive combustible substances: sulphur, charcoal, flour, sugar or oil;
 - pyrite, aluminum, zinc, cadmium and copper filings;
 - chloride salts: NH₄Cl, KCl, NaCl, BaCl₂, CaCl₂;
 - chromium, iron cations;

- inorganic acid, e.g. hydrochloric acid;
- organic impurities: animal fats, cotton, waste paper, bleaching powder, jute bags, caustic soda;
- coke, charcoal, coal, cork;
- camphor, fibers of all kinds;
- fish oil, fish meal, lubricating oil, linseed oil or drying oils, vegetable oil, naphthalene;
- hay, sawdust, and wood shavings [12].

Experimental Procedure

Materials. Ammonium nitrate used for making mixtures with various types of admixtures was N34 ammonium nitrate WE fertilizer (Polish name: *nawóz WE azotan amonu N34*) produced by Anwil Grupa ORLEN. In ammonium nitrate nitrogen occurs in two forms: nitrate and ammonia. This fertilizer was enriched with magnesium. In the initial form, it has the form of white granules (Fig. 1, 2). The total nitrogen content is 34.0 +/-0.6% (m/m).

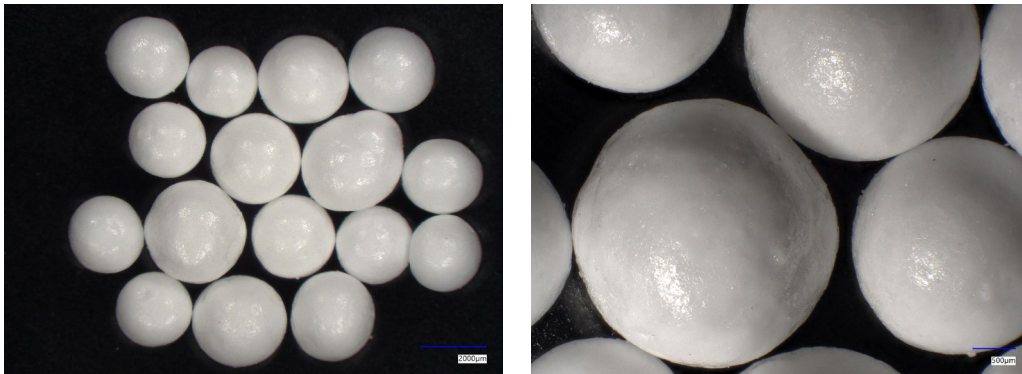


Fig. 1. Granular form of ammonium nitrate

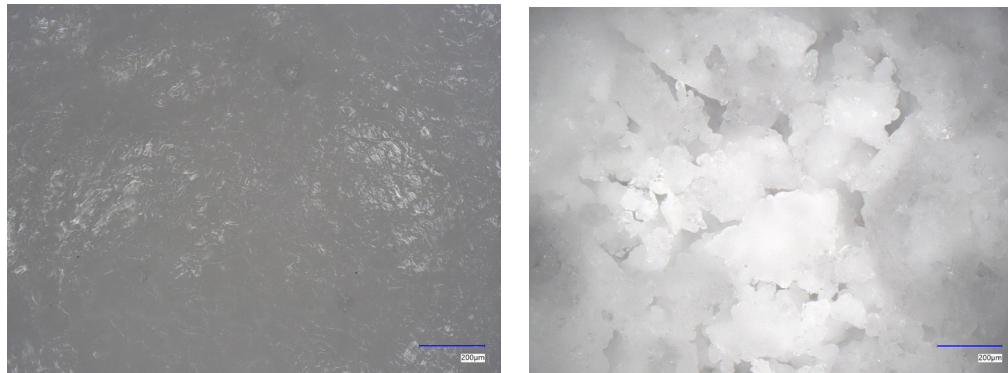


Fig. 2. Ammonium nitrate granule surface (left side) and after grinding in a mortar (right side)

Table 2. Details of sample identification

No.	Additive	Additive identification	AN%/A%	Identification of samples with AN (mixtures)
1	Pure ammonium nitrate	-	AN100	AN
2	DT0 carbon	A_1	AN80A20	Mix_1
3	Kaolin (powdered)	A_2	AN80A20	Mix_2
4	Instant coffee	A_3	AN80A20	Mix_3
5	Sand (very fine)	A_4	AN80A20	Mix_4
6	Sand (grainy)	A_5	AN80A20	Mix_5
7	Cosmetic talc	A_6	AN80A20	Mix_6
8	Face powder (loose)	A_7	AN80A20	Mix_7
9	Activated charcoal (medical)	A_8	AN80A20	Mix_8
10	Cocoa	A_9	AN80A20	Mix_9
11	Powdered sugar	A_10	AN80A20	Mix_10
12	Chalk powder	A_11	AN80A20	Mix_11

The admixture material consisted of selected materials available on the market, such as DT0 commercially available carbon (A_1), kaolin in the form of powder (A_2), instant coffee (A_3), very fine sand (dust-like) (A_4), powdered chalk, kaolin powder, sand grains, talc grains (A_5), talc (A_6), cosmetic powder (A_7), activated carbon (A_8), cocoa powder (A_9), powdered sugar (A_10) and powdered chalk (A_11). Details of the sample determination are presented in Table 2. Photographs of dopant materials are shown in Fig. 3. All the photographs with very high zoom (for impurities and AN) were taken using the KEYENCE VHX-7000N digital microscope.



Fig. 3. Optical microscope photographs of powdered and loose materials serving as admixtures in the created two-component mixtures with AN

Sample preparations and methods. Simultaneous thermal gravimetric analysis and differential scanning calorimetry (TG-DSC) were performed. The samples were analyzed using the Setline STA+ thermogravimeter by SETARAM company. Thermal analysis was performed using the Calisto software dedicated to this device. All the samples prepared were heated at the temperature

range of 30-400°C in crucibles without a lid. Samples with an initial weight of 5.0-8.5 mg were heated at 5°C.min⁻¹ under a nitrogen atmosphere.

Samples (mixtures) were prepared based on 80 wt. ammonium nitrate (AN) and 20 wt. dopant material (A). Before the preparation of two-component mixtures, the materials used were dried for min. 24 hours at 45-50°C. Before taking the samples, ammonium nitrate in the form of spheres (Fig. 1) was ground to powder (AN100) (Fig. 2). The mixtures were prepared by grinding both components in a mortar.

Results and Discussion

The purpose of the thermogravimetric measurements was to assess the thermal stability of ammonium nitrate (AN) in two-component mixtures with selected powdered or fine-grained materials. The samples were mixtures of 80 wt. AN and 20 wt. selected admixture (A). The results of DSC thermal analysis for mixtures marked Mix_1 – Mix_11 are shown in Fig.4. The values of characteristic physicochemical changes for each sample are presented in Table 3.

Table 3. The result of characteristic physicochemical transformations for each sample

Sample	Phase transition temperature [°C] (<i>onset</i>)				Endotherms [°C] (<i>onset</i>)	
	IV→III	III→II	II→I	Melting point		
AN	47.3	87.5	125.2	162.5	256.5	
Mix_1	48.3	88.0	123.2	-	146.9	
Mix_2	48.7	86.8	125.2	164.8	244.3	
Mix_3	46.1	88.4	125.4	152.8	191.2	225.5
Mix_4	45.0	87.5	125.1	162.6	249.8	
Mix_5	46.4	83.9	122.2	160.7	256.0	
Mix_6	48.8	87.2	125.2	162.6	238.8	
Mix_7	49.2	86.0	124.6	160.7	240.2	
Mix_8	51.0	88.6	124.5	-	151.7	
Mix_9	46.2	88.1	125.2	158.2	230.6	
Mix_10	46.7	87.9	100.5	-	138.2	
Mix_11	46.9	87.3	125.2	155.5	269.7	

The tests carried out using the TG-DSC method for a 100% AN sample are shown in Fig. 5. The obtained results indicate that the thermal properties of AN samples do not differ significantly from the data presented in the literature [14]. The average values of the signals obtained on the DSC curve (from 3 measurements) indicate endothermic phase transitions at temperatures of 47.3°C, 87.5°C, 125.2°C, and 162.5°C.

Similarly, as did Popławski et al. [15], in studies using differential scanning calorimetry, we obtained a signal of AN decomposition as endothermic decomposition. The authors measured AN samples in uncovered and covered crucibles (with a lid with a small hole in the middle). Both DSC curves obtained by the authors differ. When an open crucible is used, the weight loss of the sample is accompanied by the endothermic effect only. However, the use of a crucible with a lid made it impossible to remove the decomposition products of ammonium nitrate from the measurement system. This effect is clearly visible in the form of a strongly exothermic effect at temperatures above 200°C. However, in an open crucible, the TG curve confirms that the mass loss begins at a much lower temperature [15].

In the case of the tested fertilizer, the beginning of the AN decomposition exotherm starts at 181.5°C and its maximum signal is at 299.6°C (Fig. 4).

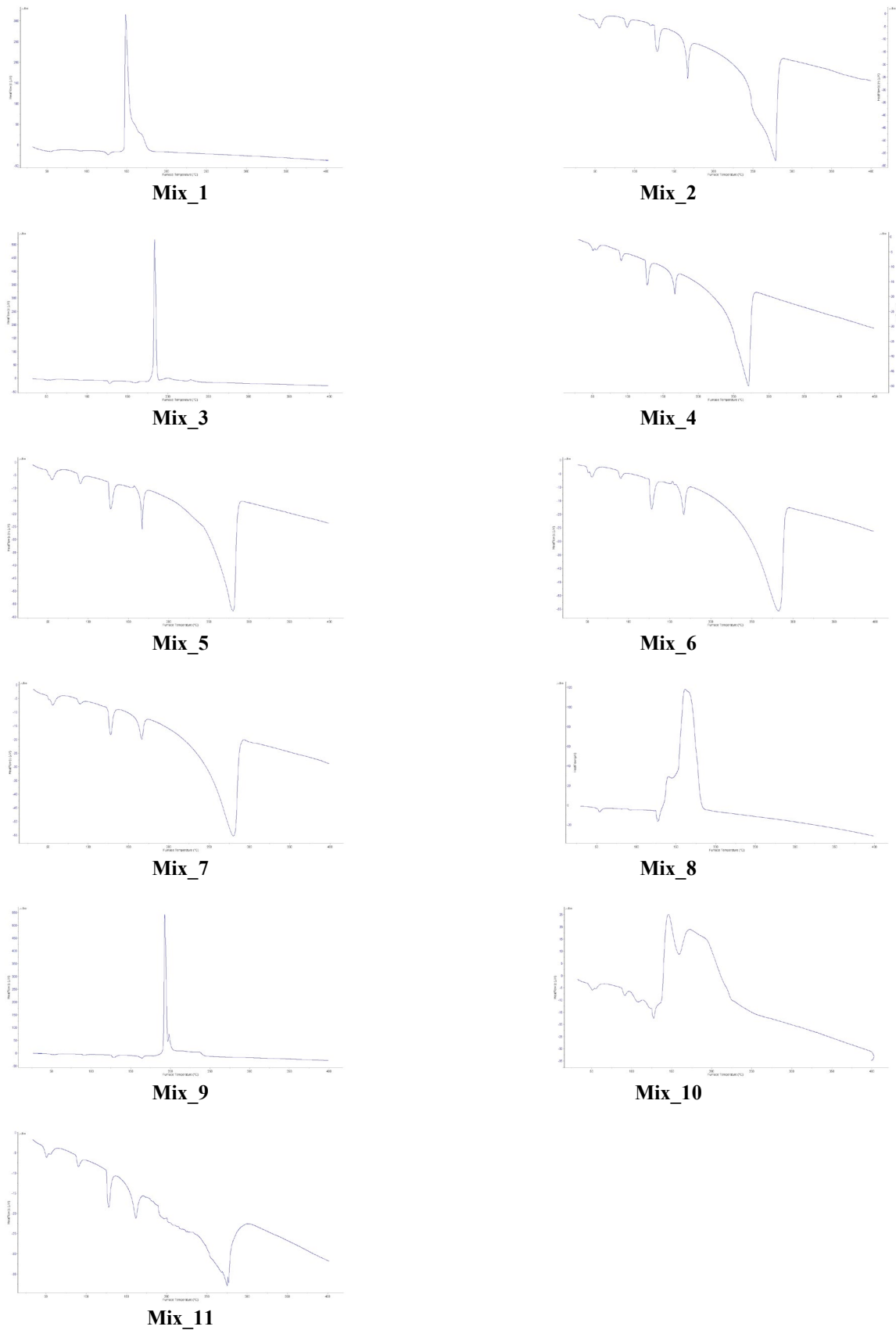


Fig.4. DSC measurement results of samples Mix_1 – Mix_11 in two-component mixtures 80 wt.% AN at 20 wt. admixtures (AN80A20)

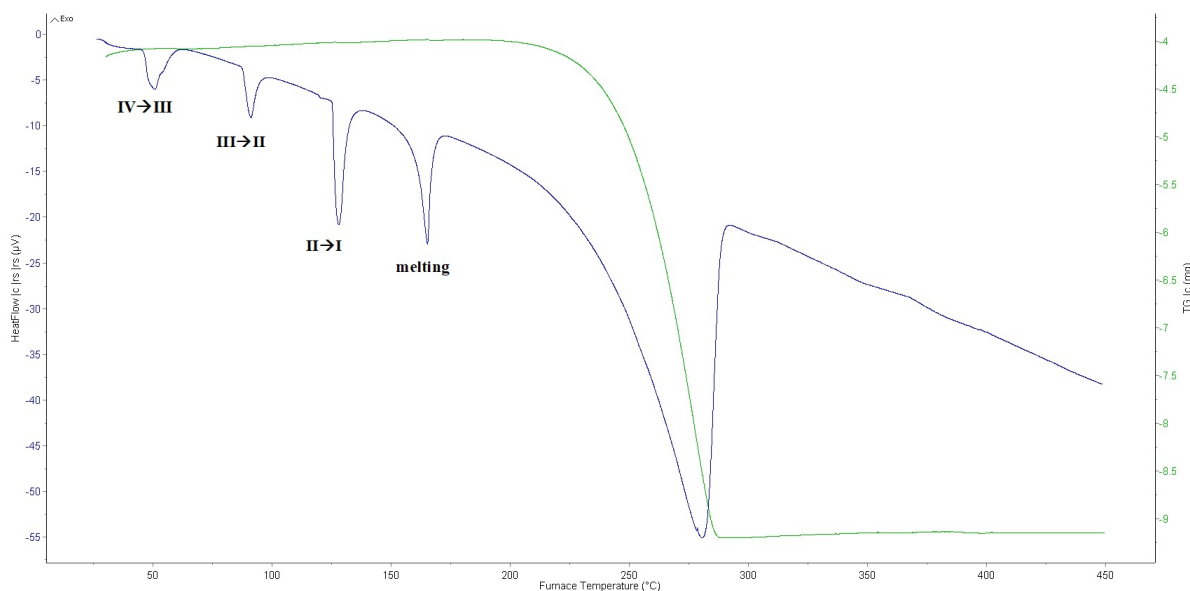


Fig.5. The results of TG-DSC measurements for non-doped AN

Based on the results of the results listed in Table 3, it should be noted that only some of the tested compounds showed the ability to reduce the thermal stability of ammonium nitrate. This effect is observed for samples labelled as Mix_1, Mix_3, Mix_8, Mix_9 and Mix_10.

In the case of samples Mix_1 (DT0), Mix_3 (instant coffee) and Mix_9 (cocoa powder) we can see a very strong decrease in the thermal stability of such mixtures. The exothermic signal observed on the DSC curves appears at 146.9°C, 181.6°C, and 190.8°C for Mix_1, Mix_3 and Mix_9, respectively. Such additives in two-component mixtures with ammonium nitrate(V) undoubtedly play the role of promoters of the analysis and reduce the thermal stability of the tested fertilizer. In the case of the Mix_3 sample, two low-intensity endothermic signals were also observed on the DSC curve, which is probably related to the successive distributions of the components contained in the sample.

In the case of mixtures of AN with powdered sugar and activated carbon (medicinal), the decomposition of samples begins at a lower temperature than the AN decomposition temperature. For Mix_8 and Mix_10, the exothermic signal splits into two vertices. For Mix_10 the reading from DSC (onset) is 138.2°C and for Mix_8 it is 151.7°C. However, this distribution is less energetic than in the case of samples Mix_1, Mix_3, and Mix_9. Therefore, powdered sugar and medicinal activated carbon decrease the stability of AN the most out of the tested mixtures. This is consistent with the data from the literature, which indicates that coal dust and organic dust are combustible components [10]. However, sugar itself is known for its effectiveness in improvised explosives [16].

Summary

The tests carried out with the use of TG-DSC made it possible to assess the effect of selected compounds on the thermal stability of ammonium nitrate. Addition of 20% (by weight) kaolin, sand regardless of its granularity, talc and cosmetic powder did not significantly affect the thermal stability of the tested samples. For the other admixtures, such as DT0 carbon, instant coffee, medicinal activated carbon, cocoa and powdered sugar (20 wt.% admixture), a shift at the beginning of the exothermic reaction accompanied by a thermal effect or a strong thermal effect was recorded. These admixtures were considered to promote decomposition, which accelerates the potential explosion of ammonium nitrate. Based on the obtained results, it can be concluded that the interaction of AN and DT0 carbon or instant coffee, medicinal activated carbon, cocoa and

powdered sugar reduces the thermal stability of the nitrate salt, as the exothermic decomposition begins at a lower temperature and is much more violent in the presence of these admixtures.

Storage and handling of explosive materials require maintaining the highest possible quality standards [17-19]. This is particularly important for ammonium nitrate, which, in its commercial form as a fertilizer [20], is not perceived as particularly hazardous, and the safety standards in agriculture are much lower than those in ammunition production [21, 22], which can pose a threat to buildings [23, 24]. The stability of explosive materials is crucial as they are also used in the metalworking industry [25-27] and alloys [28]. One can mention explosive welding as an alternative to conventional welding [29] for joining non-weldable metals, such as steel with aluminum, lead, or aluminum with copper. The resulting connections have specific surface characteristics [30], which can be a basis for their modification through the application of special coatings [31, 32], such as ESD [33, 34] or DLC [35, 36].

Further detailed examination of the mixtures mentioned in the article will require the use of special data analysis methods [37], particularly dimensionality reduction techniques [38], to avoid harmful correlations, and the implementation of statistical techniques [39-41] for identifying potential higher-order interactions, characteristic of chemical and thermomechanical problems. Non-parametric methods [42-44] and resampling techniques [45] are likely to be useful.

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