



Research paper / Praca doświadczalna

Thermal properties of modified single-base propellants Właściwości termiczne modyfikowanych prochów jednobazowych

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Abstract: The paper outlines the influence of various plasticizers (centralit I, nitroglycerine and diethylene glycol dinitrate) on the thermal properties of single-base propellants. Simultaneous differential scanning calorimetry and thermogravimetric analysis were used for this research. The basic physicochemical properties of the tested propellants were also determined, such as: geometric parameters, density and dynamic vivacity. Modifiers of the combustible layer were introduced into the pores of the nitrocellulose, based on the geometrical parameters of the propellants. The entire combustible powder layer was modified when the process was carried out in ethanol. All the modifiers used influenced the dependence of dynamic vivacity on the conversion degree. The use of 12.6 phr (part per 100 parts of propellant) of liquid nitroesters or ethanol as a dispersing medium causes a change in the propellants decomposition mechanism, which is related to a change in the structure of nitrocellulose.

Streszczenie: W pracy przedstawiono wpływ różnych plastyfikatorów (centralit I, nitrogliceryna i diazotan glikolu dietylenowego) na właściwości termiczne prochów jednobazowych. Do badań wykorzystano jednoczesną analizę skaningowej kalorymetrii różnicowej i termogravimetryczną. Określono również podstawowe właściwości fizykochemiczne badanych prochów takie jak: parametry geometryczne, gęstość i żywość dynamiczną. Modyfikatory warstwy palnej zostały wprowadzone do porów nitrocelulozy, co stwierdzono na podstawie parametrów geometrycznych prochów. Przeprowadzenie procesu w etanolu powoduje zmodyfikowanie całej warstwy palnej prochu. Wszystkie zastosowane modyfikatory wpłynęły na zależność żywości dynamicznej od stopnia przereagowania. Zastosowanie 12,6 phr (część na 100 części paliwa) ciekłych nitroestrów lub etanolu jako medium rozpraszającego powoduje zmianę mechanizmu rozkładu prochów, co związane jest ze zmianą struktury nitrocelulozy.

Keywords: modified propellants, nitrocellulose propellants, thermal properties, kinetic parameters

Słowa kluczowe: prochy modyfikowane, prochy nitrocelulozowe, właściwości termiczne, parametry kinetyczne

Symbols and abbreviations

A	pre-exponential factor [1/s]
CI	centralite I, 1,3-diethyl-1,3-diphenylurea
d_k	perforation width in the propellant grain [mm]
d_{wm}	modified layer thickness in the propellant grain [mm]
d_{wp}	combustible layer thickness in the propellant grain [mm]
D_z	propellant grain diameter [mm]
DEGDN	ethyl glycol dinitrate
DSC	differential scanning calorimetry
DTA	differential thermal analysis
E_a	activation energy [kJ/mol]
$f(\beta)$	kinetic model
HFC	heat flow calorimetry
k	reaction-rate constant [1/s]
L	propellant grain length [mm]
NC	nitrocellulose
2-NDPA	2-nitrodiphenylamine
NG	nitroglycerine
p	pressure [MPa]
p_{max}	maximum pressure [MPa]
phr	part per 100 parts of propellant
Q_0	heat of reaction [J/g]
Q_R	heat of decomposition [J/g]
Q_T	generated heat of reaction to temperature T [J/g]
R	gas constant [J/mol·K]
t	time [s]
T	temperature [°C]
T_{onset}	transition onset temperature [°C]
T_{max}	transition maximum temperature [°C]
TG	thermogravimetry
ZMW PW	Department of High-Energetic Materials, Warsaw University of Technology
α	extent of reaction expressed as pressure
β	extent of reaction expressed as heat of reaction
Γ	dynamic vivacity [1/bar·s]
Δm	mass loss [%]

1. Foreword

Due to their properties, particularly their low calorific value, high hygroscopicity and regressive burning, single-base propellants do not meet the technical requirements of modern ammunition. The regressive burning is related to the porous structure of nitrocellulose (NC) which results in the propellant deviating from the geometric burning law. The ballistic properties of the propellant can be improved by changing the structure of its combustible layer, which can be achieved by introducing by diffusion, substances known as burn rate modifiers. The modifiers can be deposited on the surface or introduced into the combustible layer, with the concentration being highest at the surface and dropping to zero at a certain depth of the propellant grain. This gradient in modifier concentration yields a propellant in which initial burn rate and gas production rate are reduced and the propellant burning progressively. The most commonly used modifiers include liquid nitrate esters, centralites, dinitrotoluene, graphite, ethylene methacrylate, aliphatic and aromatic polyols [1-4]. The ballistic parameters and the energy of single-base propellants

can be improved by introducing high-energetic components, e.g. nitroglycerine (NG), diethylene glycol dinitrate (DEGDN) or triethylene glycol dinitrate (TEGDN) into their combustible layer. Książczak *et al.* [5-7] researched the modification processes of combustible layers using different methods and different modifiers. They also used liquid nitrate esters in their studies. The modification processes were carried out using a rotary evaporator and a reactor with mechanical mixer. Water, ethyl alcohol or methylene chloride were used as the dispersing media. In most processes, other modifiers, e.g. alkyl phthalates or oligoestrols were used along with liquid nitrate esters. As a result of the study, single-base modified propellants, meeting the requirements of modern ammunition, have been obtained.

Thermal analysis is key in determining the properties of explosives, as it can be used to predict a material's behaviour in relation to its stability, compatibility, burn rate, sensitivity to impact etc. Knowledge of the thermal decomposition or kinetics of the decomposition processes can be used to develop safer explosives [8, 9]. The thermal analysis techniques commonly used in research include:

- differential scanning calorimetry (DSC),
- differential thermal calorimetry (DTA),
- thermogravimetry (TG),
- heat flow calorimetry (HFC), and
- different combined techniques, e.g. DSC-TG.

The decomposition kinetics of NC, the main component of single-base propellants, is complex due to the occurrence of parallel reactions – autocatalysis and self-heating. The thermal decomposition of NC, like other nitrate esters, begins with a homolysis of the O–NO₂ bond due to low bond energy (155 kJ/mol) [10-12]. The decomposition yields nitrogen dioxide and alkoxy derivatives (radicals). Free radicals, due to their high reactivity, immediately react with nearby ester molecules. Thus, the necessary additive in NC-based propellants includes a stabilizer which reacts with the products of NC decomposition (nitrogen oxides) interrupting or stopping the mechanism of autocatalytic decomposition. The decomposition of NC propellants in non-isothermal measurements using DSC or DTA curves is characterised by a single exothermic peak within the temperature range of 178 to 205 °C, depending on sample mass, heating rate and composition of the analysed NC propellant [13, 14]. In cases of large numbers of additives (e.g. hexogen, octogen, NG, nitroguanidine), a complex exothermic process can be observed in the DSC curves, including at least two peaks, where the second peak is related to the decomposition of the additive [15].

The purpose of the study was to analyse the thermal properties of single-base propellants with combustible layers modified with plasticizers (centralite I (CI) and liquid nitrate esters). A combined differential scanning calorimetry and thermogravimetry (DSC-TG) technique was used in the study. Basic physical and chemical properties (geometry, density and dynamic vivacity) of the propellants were also determined.

2. Experimental section

2.1. Materials used

The following materials were used in the modification process:

- 3/1 base propellant (a propellant with a 0.3 mm combustible layer and a single perforation) – Mesko S.A., Poland,
- ethanol 99.8% – Butra Skierniewice, Poland,
- distilled water,
- 2-NDPA – Department of High-Energetic Materials, Warsaw University of Technology, Poland, (ZMW PW),
- CI – Mesko S.A., Poland,
- DEGDN – ZMW PW,
- NG – Mesko S.A., Poland,
- graphite – Mesko S.A. Poland.

2.2. Base propellant modification process

The modification processes were carried out using a Heidolph Hei-VAP Advantage laboratory rotary evaporator with water bath, a G3 coiled-tube condenser cooled with a 50% aqueous ethylene glycol solution, and a Huber Minichiller, KNF Lab SC920 vacuum pump system with wireless remote control. 50 g of propellant was added to a 1 l measuring flask followed by 110 ml of dispersing medium (water or 2.5% modifier solution in ethanol). The mixture was heated to 70 °C, 5% modifier solution in ethanol was added (if water was used as a dispersing medium) or water (if ethanol was used as a dispersing medium) at a specific rate (1 ml every 5 min). The modified layer thickness indicator (2-NDPA) was added with the modifier solution in ethanol in the amount of 0.2 part per 100 parts of propellant (phr). After adding the modifier or water, the ethanol was distilled under reduced pressure. The filtered propellant was graphite coated using 0.2 phr graphite. The graphite-coated propellant was dried under forced air circulation to constant mass. Table 1 shows the obtained propellants.

Table 1. The obtained propellants as a result of modification

Propellant	Dispersing medium	Modifier	
		type	Quantity in phr
M1	water	CI	3
M2		DEGDN	
M3	ethanol		
M4	water	DEGDN and NG at 1:2 ratio	12.6
M5		NG	

2.3. Test methods

The geometries of the propellant grains were determined using a Delta Optical Smart 5MP PRO optical microscope. 100 propellant grains were selected at random and measured (length L and width D_z). The sections of another 50 randomly selected grains were prepared to determine the perforation width (d_k), combustible layer thickness (d_{wp}) and modified layer thickness (d_{wm}) by adding 2-NDPA with a characteristic red and orange colour.

The density of the propellants was determined using a Micrometrics AccuPyc 1330 helium pycnometer. All measurements were made at room temperature (25 °C). A total of 30 measurements were made for each propellant to calculate the averages and standard deviations.

The dynamic vivacity of the propellants was calculated based on measurements carried out using a BT-25/500 manometric bomb. Propellant with a mass of 4.0000 ± 0.0005 g was used. The propellant was ignited with a primer (200 mg gunpowder) and the change of pressure with time was recorded. The dynamic vivacity of the propellant (gas production rate) was calculated from the following equation (assuming that the propellant burned in layers at a constant rate) [16]:

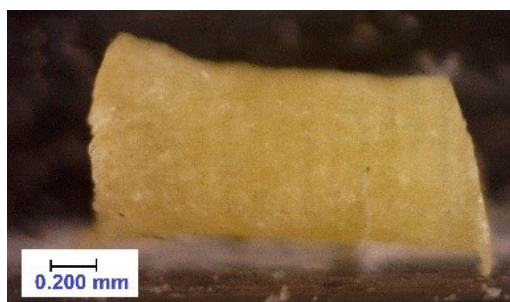
$$\Gamma = \frac{d\alpha}{dt} \cdot \frac{1}{p} \quad (1)$$

where: Γ – dynamic vivacity, α – extent of reaction (the amount of propellant burned), da/dt – gas production rate, p – pressure. The extent of reaction is expressed as a ratio of pressure (p) to maximum pressure (p_{max}). The thermal properties of the propellants were determined using an instrument which combined differential scanning calorimetry and thermogravimetry (DSC-TG), a SDT Q600 by TA Instruments. The measurements were carried out in open aluminium containers containing 3.2 to 4.1 mg samples (three propellant grains were placed in each container). The measurements were carried out at 30 to 300 °C at a temperature increase rate of 1 °C/min in a nitrogen atmosphere (100 ml/min).

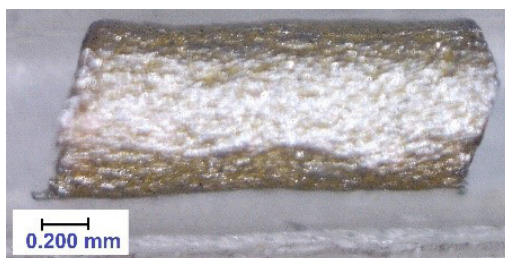
3. Results and discussion

3.1. Determination of geometric parameters

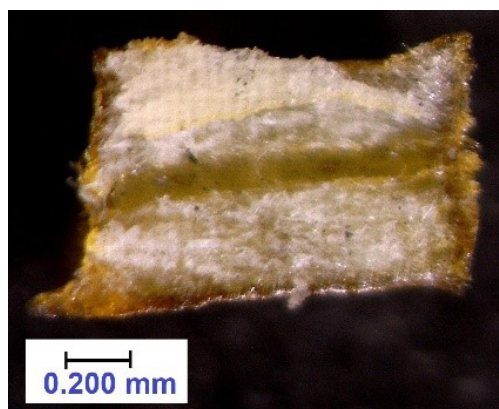
Before analysing the thermal properties of the NC propellants, basic physical and chemical tests were carried out, including the analysis of geometrical parameters, density and dynamic vivacity of the propellant grains. To determine the effect of modification on grain geometry, randomly selected grains were analysed and the geometric parameters were determined, see Table 2. Figure 1 shows sample propellant grains before and after modification (M2 propellant).



(a)



(b)



(c)

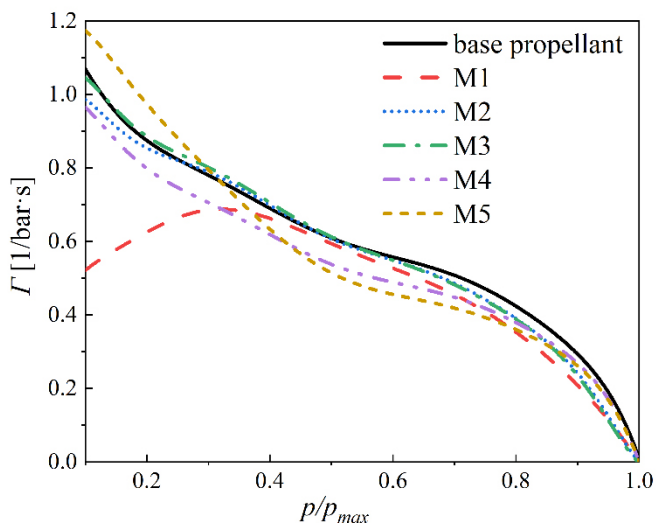
Figure 1. Propellant grains before modification (a), after modification – M2 propellant – full grain (b) and grain cut in half (c)

Table 2. Geometric parameters and densities of modified propellants

Propellant	L [mm]	D_z [mm]	d_{wp} [mm]	d_k [mm]	d_{wm} [mm]	Density [g/cm ³]
Base	1.68 ±0.31	0.74 ±0.04	0.31 ±0.05	0.12 ±0.02	–	1.623 ±0.004
M1	1.57 ±0.23	0.72 ±0.03	0.30 ±0.05	0.13 ±0.02	0.03 ±0.01	1.622 ±0.002
M2	1.53 ±0.28	0.72 ±0.04	0.30 ±0.05	0.12 ±0.02	0.06 ±0.01	1.631 ±0.003
M3	1.53 ±0.23	0.72 ±0.02	0.29 ±0.05	0.13 ±0.02	0.29 ±0.05	1.658 ±0.002
M4	1.55 ±0.28	0.73 ±0.04	0.31 ±0.04	0.12 ±0.02	0.08 ±0.01	1.636 ±0.002
M5	1.54 ±0.23	0.74 ±0.04	0.31 ±0.04	0.13 ±0.02	0.07 ±0.01	1.646 ±0.002

The results show that the modification process does not affect the geometric parameters of the propellant grains (allowing for measurement error) which means that the modifiers have been introduced into the NC pores. Using ethanol as a dispersing medium allowed the modifier to be distributed over the entire thickness of the combustible layer (M3 propellant). For other propellants, the modified layer thickness was 20-26% of the combustible layer thickness. An increase in the amount of nitrate esters also increases the modified layer thickness, however, the difference is minor – by 10-20 μm for M4 and M5 propellants (12.6 phr nitrate ester) compared to M3 propellant (3 phr nitrate ester). The density of M1 propellant is similar to that of the base propellant, whereas other propellants show increased density compared to the base propellant. The highest density was observed for M3 propellant modified in ethanol.

Figure 2 shows the dynamic vivacity of the base propellant and the modified propellants. The burning process of all propellants is regressive – the burning surface area decreases with time; the only exception is the M1 propellant which at the initial stage (up to $p/p_{max} \approx 0.35$) shows progressive burning, shifting to regressive burning. M5 propellant shows the highest dynamic vivacity at $p/p_{max} = 0.32$. M2 and M3 propellants modified with 3 phr DEGDN showed a similar relationship to the base propellant. The results show that the type of dispersing medium does not affect the rate of gas production. A decrease in dynamic vivacity, compared to the base product, was observed for M4 propellant modified with a mixture of liquid nitrate esters.

**Figure 2.** Comparison of the dynamic vivacities of the base propellant and modified propellants

3.2. Determination of thermal properties of the propellants

The thermal properties of the propellants were determined using DSC-TG analysis. Figure 3 is an example of a DSC-TG curve for M1 propellant. The DSC curve shows a single exothermic peak related to the thermal decomposition of the propellant. The decomposition process is accompanied by a single stage loss of mass. The DSC-TG curves are similar for all analysed propellant samples. Based on the results, the following parameters were determined: onset temperature (T_{onset}), maximum temperature (T_{max}), heat of decomposition (Q_{R}) and loss of mass (Δm), see Table 3.

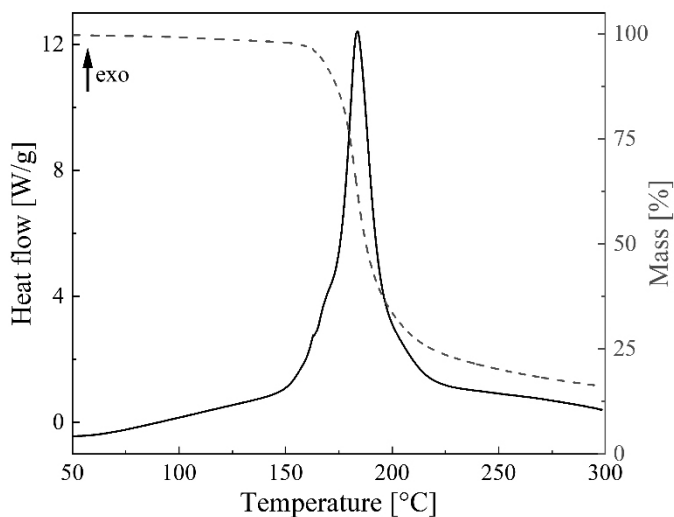


Figure 3. DSC-TG curve for M1 propellant

Table 3. Parameters determined based on the DSC-TG curves

Sample	T_{onset} [°C]	T_{max} [°C]	Q_{R} [J/g]	Δm [%]
Base propellant	172.25	183.67	3757	82.15
M1	172.65	183.90	3737	81.28
M2	173.37	183.69	3646	81.67
M3	173.52	184.34	3525	83.09
M4	171.65	183.78	3618	81.67
M5	172.52	183.58	3468	81.26

Interpretation of the results shows that the onset and maximum temperatures are not affected by the type and amount of modifier used. The differences between the measurements fall within the range of instrument error. The average onset temperature is 172.7 ± 0.7 °C, and the average maximum temperature is 183.8 ± 0.3 °C. The heat of decomposition of the propellant modified with 3 phr CI (M1) is similar to the heat of decomposition of the base propellant. For other propellants modified with liquid nitrate esters (M2 to M5), the heat of decomposition is lower than for the base propellant. Similarly, the loss of mass determined at temperatures between 100 and 280 °C was not affected by the type and amount of modifier used – except for M3 propellant (modified with 3 phr DEGDN in alcohol medium), where the loss of mass was the highest at 83%. The average loss of mass for the other propellants was $81.6 \pm 0.4\%$.

The kinetics of NC propellant decomposition can be determined using DSC [17]. The extent of reaction (β) of the material is calculated based on the ratio of generated heat of reaction to temperature T (Q_{T}) to the total heat of reaction (Q_0):

$$\beta = \frac{Q_T}{Q_0} \quad (2)$$

The rate of decomposition reaction can be expressed as a general kinetic equation:

$$\frac{d\beta}{dt} = k \cdot f(\beta) \quad (3)$$

where: k – reaction rate constant, t – time, $f(\beta)$ – kinetic model.

The relationship between the reaction rate constant and temperature can be expressed as an Arrhenius equation:

$$k = A \cdot e^{\frac{-E_a}{RT}} \quad (4)$$

where: A – pre-exponential factor, E_a – activation energy, R – gas constant.

Assuming a zero-order kinetic model $\left(k = \frac{d\beta}{dt}\right)$, the following relationship can be obtained by substituting Equation 2 into 3:

$$k = \frac{d\beta}{dt} = A \cdot e^{\frac{-E_a}{RT}} \quad (5)$$

The logarithm of Equation 4 is calculated to determine the kinetic parameters (A and E_a):

$$\ln\left(\frac{d\beta}{dt}\right) = \ln(A) - \frac{E_a}{RT} \quad (6)$$

Based on the relationship $\ln\left(\frac{d\beta}{dt}\right)$ of the reciprocal temperature, a linear relationship is obtained in which the kinetic parameters are determined by its slope and absolute term after transformations. To determine the relationship between the NC structure in propellants and the kinetics of decomposition, a kinetic analysis of an area in which the decomposition of NC is the main process in relation to the decomposition of the products of the process, must be carried out. Thus, kinetic analysis was carried out to determine the extent of reaction between 0.01 and 0.50. Figure 4 shows a typical kinetic curve showing different decomposition stages and the DSC curve for M2 propellant. The kinetic curve includes three areas related to heterogeneity of the structure in the entire volume of the propellant:

- **first area:** related to the kinetics of decomposition of the outer layer of the propellant; the decomposition starts in the closed pores,
- **second area:** related to the evaporation of low molecular weight components of the propellant, corresponding to a minor change in the slope of the DSC curve,
- **third area:** related to the kinetics of decomposition of the inner layers of the propellant; the process takes place in the open pores.

The stages of propellant decomposition were verified in a study carried out by Zygmunt *et al.* [17] by verifying the kinetics of decomposition of the propellant conditioned for 3 h at increased temperature to eliminate the closed pore structure and the propellant pressed under 0.9 GPa pressure to eliminate the porous structure. Both propellants, prepared using this method showed a uniform decomposition mechanism without identifiable stages.

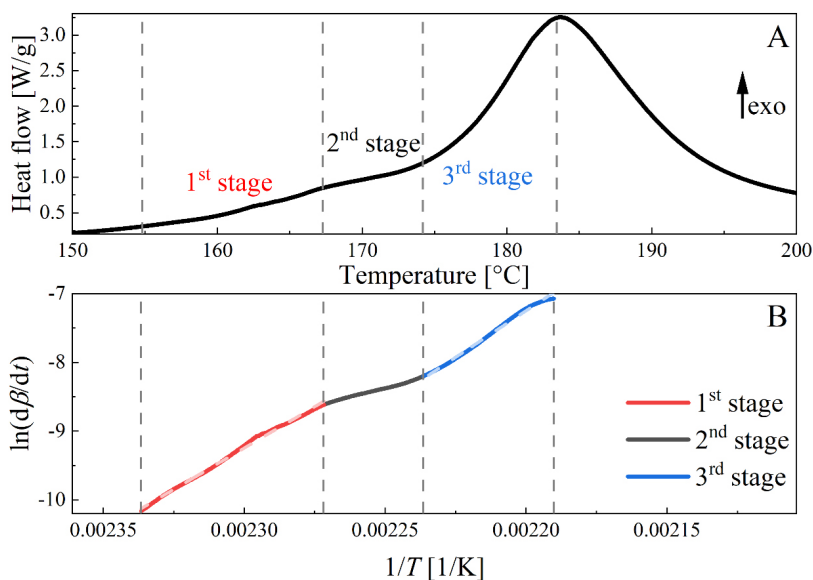


Figure 4. DSC (A) and kinetic (B) curves for M2 propellant with identifiable decomposition stages

Stages 1 and 3 were defined using a linear equation (dashed lines in Figure 4B), based on which the kinetic parameters E_a and $\ln A$ were determined using Equation 5. Table 4 shows the parameters determined for the analysed propellants.

Table 4. Kinetic parameters determined based on the kinetic curve

Sample	Stage 1		Stage 3	
	E_a [kJ/mol]	$\ln A$	E_a [kJ/mol]	$\ln A$
Base propellant	200.1 ± 0.3	46.2 ± 0.1	196.6 ± 0.6	44.7 ± 0.2
M1	219.7 ± 0.4	51.6 ± 0.1	200.3 ± 0.6	45.7 ± 0.1
M2	199.9 ± 0.4	46.1 ± 0.1	224.8 ± 0.6	52.2 ± 0.2
M3	181.9 ± 0.2	40.8 ± 0.1	–	–
M4	176.4 ± 0.2	39.5 ± 0.1	–	–
M5	174.8 ± 0.2	39.0 ± 0.1	–	–

For M3-M5 propellants, the kinetic parameters were determined for the first stage only, since no stages related to different NC decomposition mechanism were observed in the kinetic curve. The propellants were modified with 3 phr DEGDN in alcohol medium (M3) and an increased amount of nitrate ester or a mixture of nitrate esters – 12.6 phr (M4-5). This means that both the modification in alcohol medium and a four-fold increase in added modifier, significantly change the porous structure of the propellant. The use of energetic plasticizers turns NC into gel which significantly reduces the number of pores which are filled with the introduced low molecular weight components. The activation energy observed in the first stage for M3-M5 propellants is approx. 20 to 25 kJ/mol lower compared to the base propellant – caused by the amount of nitrate esters used which reduce the thermal stability of the propellants. It is also worth noting that M2 propellant contains the same amount of DEGDN as M3 propellant but uses a different method of introducing the modifier in a different dispersing medium (M2 – water, M3 – ethanol) and shows the activation energy in the first stage similar to the base propellant. It means that the distribution of the modifier within the NC structure, significantly affects the kinetic parameters, and hence the thermal

stability of the propellant. As expected, M1 propellant shows the highest activation energy in the first stage due to the use of 3 phr CI which functions both as a stabilizer and a plasticizer for NC. The activation energies observed in the third stage are lower than the activation energies observed in the first stage for M1 and M2 propellant, whereas the reverse has been observed for M3 propellant.

4. Summary

- ◆ Five modification processes were carried out using the following combustible layer modifiers: CI, DEGDN and NG, as a result of which, the geometric parameters within the standard deviation range have not changed compared to the base propellant which means that the modifiers had been introduced into the NC pores.
- ◆ Using ethanol as a dispersing medium modifies the entire combustible layer of the propellant. When water is used in the modification processes, the modified layer thickness is between 20% and 26%. Modifying the propellant with 3 phr CI did not affect the density compared to the base propellant, as opposed to other modifications which resulted in an increase in density. The dynamic vivacity of the propellants modified with DEGDN was not significantly affected compared to the base propellant. No effect of the dispersing medium used on the dynamic vivacity of the analysed propellants has been observed.
- ◆ Using a mixture of liquid nitrate esters decreased the dynamic vivacity compared to the base propellant. Modification with 3 phr CI affected the nature of the burning process compared to the base propellant. The applicability of the test propellants will be verified following a graphite coating process, and ballistic tests will be carried out to determine those parameters which are significant for different ammunition types.
- ◆ A DSC-TG analysis was used to determine the kinetic parameters of the decomposition process of the obtained modified propellants. Three stages related to the heterogeneity of propellant structure and affecting the type of the decomposition mechanism, can be identified in the kinetic curve. Using ethanol as a dispersing medium or a large amount of liquid nitrate esters (12.6 phr) changes the mechanism of decomposition – single decomposition stage can be identified in the kinetic curve. It is related to the degree in which NC turns into gel and the distribution of the modifiers in the combustible layer of the modified propellants.
- ◆ The activation energy of the propellants showing a single-stage decomposition mechanism was 20 to 25 kJ/mol lower than for the base propellant. The highest activation energy of the decomposition process was observed in the propellant modified with 3 phr CI which functions both as a stabilizer and a plasticizer.

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