

BURNING BEHAVIOUR OF ADN SOLID PROPELLANTS IN COMPARISON TO OTHER OXIDIZERS

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ABSTRACT

Solid rocket propellants based on ammonium dinitramide (ADN) as oxidizer and glycidyl azide polymer (GAP) as binder are suitable candidates for future propellants with good performance characteristics and no hazardous impact to the environment relating to the burning products. The comparison with the actual state of the art solid propellant, based on hydroxyl-terminated polybutadiene (HTPB) and ammonium perchlorate (AP), reveals that ADN-based propellants, especially in combination with an energetic binder, result in non-suitable burning rates (rb) and pressure sensitivities (n) for civil applications. Well known ballistic modifier to reveal an optimized burning behaviour with the main focus on rb and n are inefficient, incompatible to the oxidizer (ADN) or generate undesirable burning phenomena.

For this reason, propellant formulations of ADN/GAP, ADN/HTPB and AP/GAP, AP/HTPB were produced and investigated to comprehend the differences in the combustion mechanism. The characterization of the burning behavior was done in a chimney-type window bomb (2 – 13 MPa). By the burning interruption of the propellants in liquid nitrogen, the reaction layer near the surface of the different sample formulations is analyzed in terms of Nano-Computed Tomography (nano-CT) and Confocal Raman Spectroscopy.

Keywords: Combustion behaviour, Solid rocket propellants, ADN, AP

1 Introduction

Solid rocket propellants typically consist of a mixture of granules of a solid oxidizer (AP, AN, ADN) placed in a polymeric binder combined with energetic compounds (HMX, RDX), metallic additives (Al, Mg) plasticizers, stabilizers and / or burn rate modifiers [1].

Today, HTPB (hydroxyl-terminated polybutadiene, binder) and AP (ammonium perchlorate, oxidizer) are widely used in solid propellants. These propellants are well known for their good performance characteristics, mechanical properties and the wide operating temperature range. But the contamination of the environment by the combustion products of perchlorate is also well known and documented [2]. For this reason, the future propellants should keep the performance characteristics and the mechanical properties, but replace the components, which lead through the burning to a hazardous contamination of the environment. One oxidizer, which has the potential to fulfil the criteria is ADN. In combination with non-energetic binders like HTPB, similar specific impulse values comparable to systems based on HTPB/AP could be reached. Pressure exponents higher than 0.7 for HTPB/ADN formulations [3] make them invalidate for the majority of potential applications. Another approach is the usage of GAP as an energetic binder. GAP is classified as a high-nitrogen content polymer and due to its availability, good binder properties and low detonation sensitivity it is a suitable polymer for solid propellants [4], [5]. Usually, GAP/ADN propellant formulations, which are reaching similar specific impulse values as comparable systems based on HTPB/AP, feature a very high burning rate in the operation range between 2 and 13 MPa. The pressure exponent is typically ranging from 0.3 to 0.7 [6], [7], [8]. ADN-based propellants in combination with an energetic binder, result in non-suitable burning rates for civil applications.

In order to optimize the properties of an ADN-based propellant, burning rate modifiers are needed. For this reason, propellant formulations of GAP/ADN, HTPB/ADN and GAP/AP, HTPB/AP were investigated to assess the differences in the combustion mechanism.

2 Experimental Procedure

2.1 Materials

In summary, eight formulations of solid composite propellants have been investigated. The main differences are the used oxidizer and binder, with in summary three different adjusted oxidizer/binder ratios (Table 1).

Table 1 Composition of the investigated solid propellants with different oxidizers and binders.

Labelling	HTPB	GAP [%]	ADN [%]	AP [%]	Ratio Oxidizer/Binder
HADN_21_79	21.0		79.0		3.76
GADN_27_73		27.0	73.0		2.70
HADN_27_73	27.0		73.0		2.70
GADN_43_57		43.0	57.0		1.33
HAP_21_79	21.0			79.0	3.76
GAP_27_73		27.0		73.0	2.70
HAP_27_73	27.0			73.0	2.70
GAP_43_57		43.0		57.0	1.33

The labelling of the samples is chosen that the important information is immediately identifiable. The first letter stands for the used binder (H=HTPB; G=GAP). The further letters label the oxidizer (ADN, AP), followed by the mass percentage of binder (first number) and oxidizer (second number) of the investigated formulations.

The produced solid composite propellants include ADN or AP prills with particle sizes of 176 μm (ADN) or 200 μm (AP). The selected curing system for the energetic binder (GAP) is a combination of three isocyanates (Desmodur N 100, Desmodur N 3400, and Desmodur XP 2617) for a comprehensive NCO/OH ratio of 0.9. Increasing the reaction rate, the catalyst dibutyltin dilaurate (D22) was added. For the curing of the non-energetic binder formulations, one isocyanate (IPDI) and the same catalyst (DBTL, D22) were used. Overviews of the components are listed in Table 2.

Table 2 Chemicals used in experimental work.

Component	Class	Supplier
ADN	Oxidizer	Synthesized at EUB, prilled at FOI and ICT
AP	Oxidizer	SNPE, Japan
GAP 06S12	Binder	Eurenco, Sweden
Polyvest EP HT	Binder	Evonik, Germany
Alcan 400	Metal Fuel	Krahn Chemie, Germany
Desmodur N 100 / 3400	Curing Agent	Covestro AG, Germany
Desmodur XP 2617	Curing Agent	Covestro AG, Germany
IPDI	Curing Agent	Evonik, Germany
Dibutyltin dilaurate (DBTL, D22)	Catalyst	Merck, Germany

All the propellants discussed in this paper were produced with the ARV-310 Thinky Mixer, a planetary centrifugal bladeless kneader, “under vacuum” (~1000 Pa), in order to minimize air inclusions induced by the mixing process in the slurries.

2.2 Thermodynamic Calculations

The formulations were selected in order to compare the different oxidizer – binder combinations due to similar values of the specific impulse (I_{sp}) and three fixed oxidizer/binder ratios. Matching the range of the world spread HTPB/AP/Al (68/14/18) propellant specific impulse (I_{sp}) of 266 s was not in the main focus. The thermodynamic calculations were performed with ICT Thermodynamic Code [9] under shifting equilibrium hypothesis and pressure ratio of 70:1.

As can be seen in Table 3 and Figure 1 the used propellants achieving a specific impulse range of 199 s – 257 s, depending on the used oxidizer (AP, ADN) and the ratio of oxidizer and binder.

In a first batch, four different oxidizer/binder (GAP/ADN, GAP/AP, HTPB/ADN, HTPB/AP) combinations were produced with the same oxidizer/binder ratio of 2.70. The formulation of GAP/ADN with 27/73 has the highest calculated specific impulse of 257 s. The choice of the binder has a more distinct influence on the I_{sp} than the oxidizer. The formulation HTPB/ADN with 27/73 reach an I_{sp} of 203 s, which is significantly lower than the GAP/AP combination with 27/73 (251 s).

In a second batch, formulations should be produced with a similar calculated specific impulse in the range of 230 s. The limiting factor was the processability for some propellants, especially the combination of oxidizers with the non-energetic binder HTPB. Formulations of HTPB/AP (21/79) exhibit the highest derivation to the target

with 226 s. With a specific impulse of 229 s (HTPB/ADN) and 230 s (GAP/ADN) the ADN-based propellants reach the demanded I_{sp} .

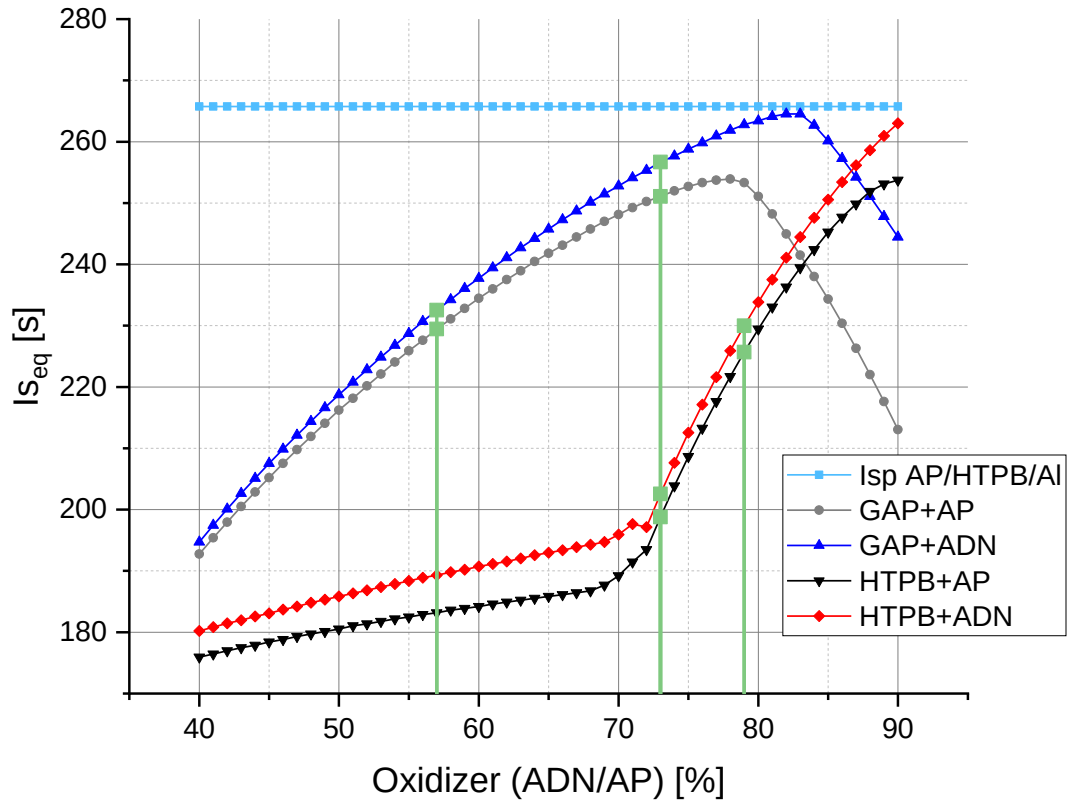


Figure 1 Specific impulse from thermodynamic calculations for the oxidizer/binder combinations: GAP/AP, GAP/ADN, HTPB/AP and HTPB/ADN (comp. Table 3).

Table 3 Thermodynamic calculations of the specific impulse (I_{sp}) for the oxidizer/binder combinations: GAP/AP, GAP/ADN, HTPB/AP and HTPB/ADN.

Labelling	Ratio Oxidizer/Binder	Isp [s]
HADN_21_79	3.76	229.97
GADN_27_73	2.70	256.68
HADN_27_73	2.70	202.55
GADN_43_57	1.33	232.52
HAP_21_79	3.76	225.69
GAP_27_73	2.70	251.07
HAP_27_73	2.70	198.78
GAP_43_57	1.33	229.46

The adiabatic combustion temperature (T_{ad}) is a further important parameter for the combustion behaviour, which was calculated by the ICT Thermodynamic Code (Figure 2 and Table 4). As to be expected, the composition with the highest amount of oxidizer (AP, ADN) in combination with the energetic binder GAP reaches the maximum calculated T_{ad} with 3036 K (GAP/AP) and 2943 K (GAP/ADN). Formulations with a low oxidizer content in combination with the non-energetic binder HTPB reveal the minimum calculated T_{ad} with 1577 K (HTPB/ADN) and 1632 K (HTPB/AP). Considering a constant oxidizer/binder ratio, the change from an energetic binder (GAP) to a non-energetic (HTPB) is leading to a dramatic decrease of the adiabatic combustion temperature of more than 1500 K.

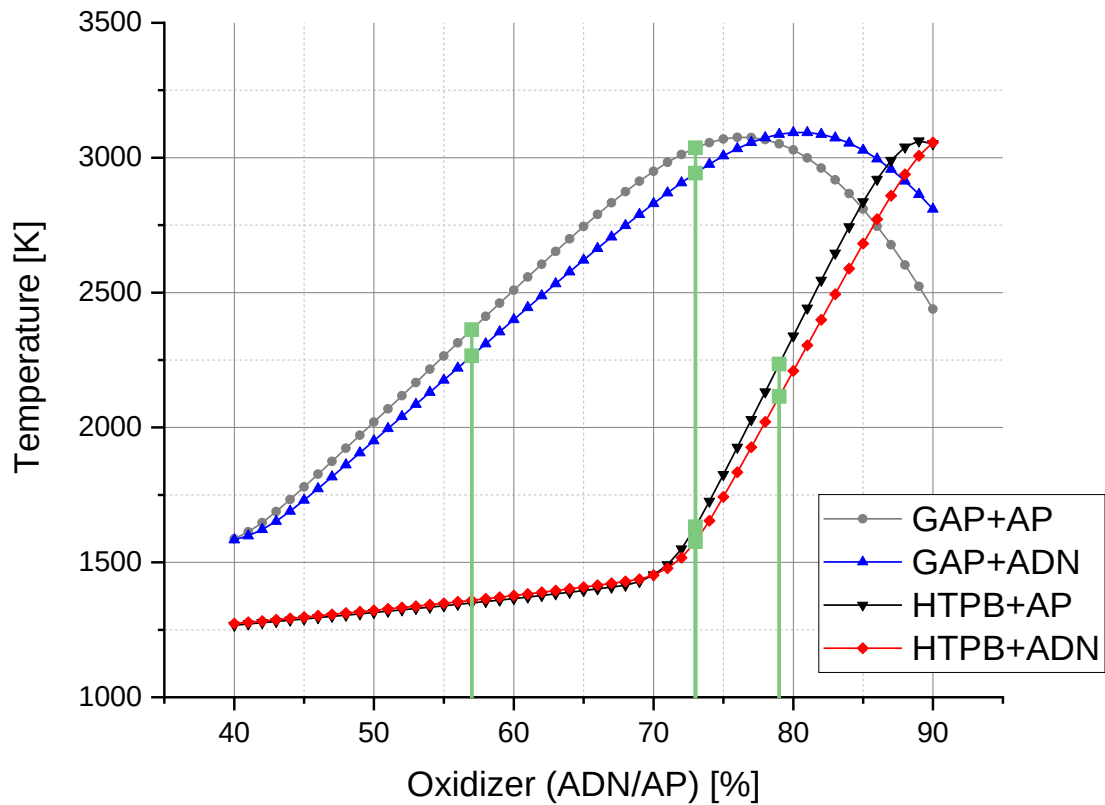


Figure 2 Adiabatic combustion temperature from thermodynamic calculations for the oxidizer/binder combinations: GAP/AP, GAP/ADN, HTPB/AP and HTPB/ADN (comp. Table 4).

Table 4 Thermodynamic calculations of the adiabatic combustion temperature (T_{ad}) for the oxidizer/binder combinations: GAP/AP, GAP/ADN, HTPB/AP and HTPB/ADN.

Labelling	Ratio Oxidizer / Binder	Temperature [K]
HADN_21_79	3.76	2114.70
GADN_27_73	2.70	2942.70
HADN_27_73	2.70	1576.50
GADN_43_57	1.33	2265.30
HAP_21_79	3.76	2235.40
GAP_27_73	2.70	3036.80
HAP_27_73	2.70	1631.90
GAP_43_57	1.33	2362.80

2.3 Experimental Setup

For the general characterization, samples were tested as strands in a window bomb at 2, 4, 7, 10 and 13 MPa nitrogen pressure. In the window bomb the burning rate was measured by a colour high-speed video camera using an improved computer analysing technique, which allows to reduce the sample size and to exclude failed tests. The emission spectra were captured by an UV/Vis/NIR spectrometer. The emission temperatures were derived from the spectra by using the ICT-BaM software, as described elsewhere [10]. The samples were ignited with a melting wire placed at the top end. Every sample was measured at least twice.

In order to get a better understanding of the burning mechanism for each propellant, the samples were forcibly extinguished in the window bomb. Relating to this, the fast depressurization technique [11] was ineffective due to the low pressure deflagration limit (PDL) of GAP/ADN formulations (~ 0.015 MPa), and a new method has been developed. The strand is held over a polystyrene insulated test tube filled with liquid nitrogen by a thin wire, in turn fixed to the structure. The whole assembly, depicted in Figure 3, is introduced into the window bomb. When ignited, as soon as the burning surface approaches the wire, the sample falls into liquid nitrogen and the combustion is quenched. The vessel pressure is to be chosen in such a way that the strand ignites and burns uniformly and, at the same time, it is instantaneously quenched when submerged into liquid N_2 ; for this reason, values near the PDL were typically chosen.

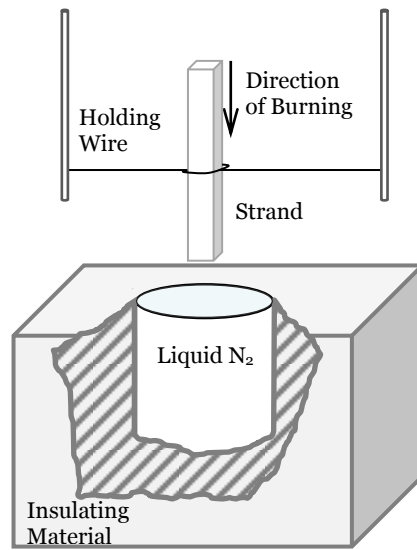


Figure 3 Schematic of experimental setup for forced extinction.

Finally, Nano Computed Tomography (Nano-CT) measurements and Confocal Raman microscope has been employed to examine the structure of the extinguished samples.

3 Results and Discussion

To compare the strand burn results in dependence of the pressure, the produced propellants were summarized in two groups. In the first one, ADN was chosen as oxidizer in combination with the binders HTPB and GAP with three different oxidizer/binder ratios. The results are shown in Figure 4 and Table 5.

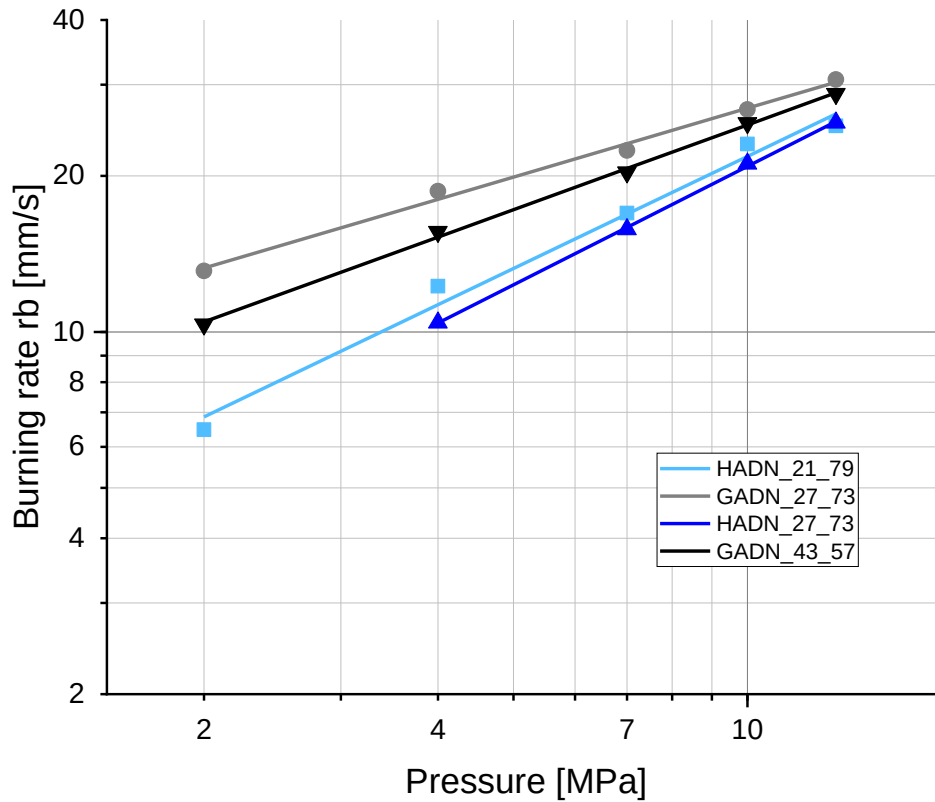


Figure 4 Influence of the binder-type and the oxidizer/binder ratio on the burning behaviour of ADN-based propellant formulations.

Table 5 Influence of the binder-type and the oxidizer/binder ratio on the burning rate at 7 MPa of ADN-based propellants and overview of the calculated pressure exponent (n).

Labelling	Ratio Oxidizer / Binder	r_b (7 MPa) [mm/s]	Pressure exponent (n)
HADN_21_79	3.76	16.95	0.74
GADN_27_73	2.70	22.39	0.44
HADN_27_73	2.70	15.78	0.76
GADN_43_57	1.33	20.34	0.52

In general, all measured samples show a burning behaviour which can be described with high accordance by the Vieille's law equation:

$$r_b = A \cdot \left(\frac{p}{p_0}\right)^n \quad (1)$$

In combination with HTPB as a non-energetic binder, ADN based propellants show the highest pressure exponent, independent of the adjusted oxidizer/binder ratio with $n > 0.7$. The lowest pressure exponent was reached with the formulation GAP/ADN with 27 wt % of binder ($n = 0.44$). Compared to GAP/ADN formulations, the measured burning rates for HTPB/ADN are invariably lower at every pressure step. At 7 MPa, the difference in r_b for HTPB and GAP propellants with the same oxidizer/binder ratio (2.70) is 6.61 s ($r_b(\text{HTPB/ADN}) = 22.39$ s; $r_b(\text{GAP/ADN}) = 15.78$ s). As expected, the burning rate is decreasing with reducing the total amount of oxidizer in the propellant formulation. With the exception of the HTPB/ADN formulation 27/73, all samples could be ignited at 2 MPa.

In a second step, the AP-based formulations with different binders were compared. The results of the burning rate measurement in dependence of pressure are shown in Figure 5 and Table 6.

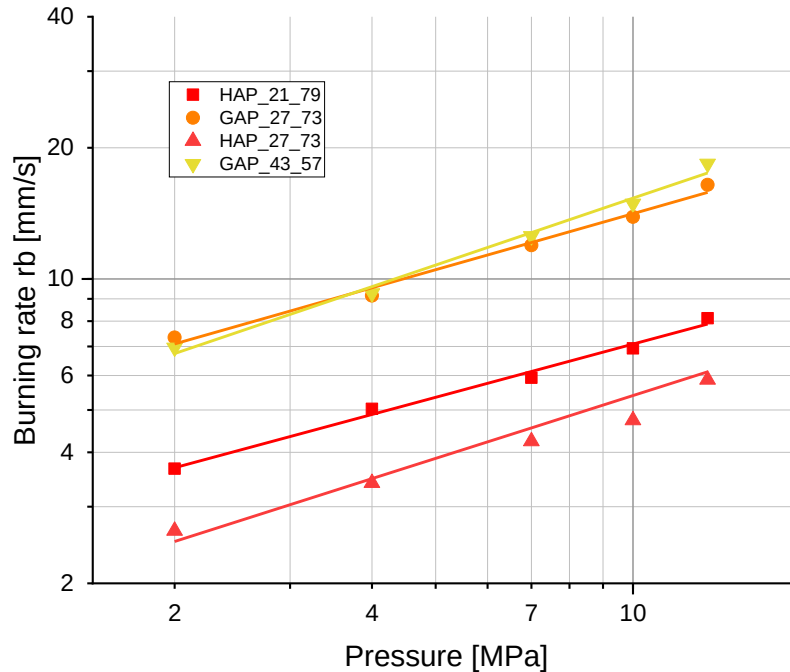


Figure 5 Influence of the binder type and the oxidizer/binder ratio on the burning behaviour of AP-based propellant formulations.

Table 6 Influence of the binder type and the oxidizer/binder ratio on the burning rate at 7 MPa of AP-based propellants and overview of the calculated pressure exponent (n).

Labelling	Ratio Oxidizer / Binder	r_b (7 MPa) [mm/s]	Pressure exponent (n)
HAP_21_79	3.76	5.93	0.41
GAP_27_73	2.70	11.94	0.43
HAP_27_73	2.70	4.24	0.48
GAP_43_57	1.33	12.54	0.52

In contrast to ADN, the binder selection does not give the main influence on the pressure exponent. HTPB- and GAP-based samples with the same oxidizer/binder ratio (2.7) show similar values for n ($n(\text{GAP}_{27_73}) = 0.428$; $n(\text{HAP}_{27_73}) = 0.479$), even when the burning rate at 7 MPa is more than doubled for GAP-based propellants compared to HTPB. For HTPB-based propellants in combination with AP, a small increase in the total amount of oxidizer (+ 7.6 wt %) results in a significant change in the burning rate at each pressure step of more than +25 %. On the other hand, for GAP-based formulations, the oxidizer/binder ratio has apparently no dramatic effect regarding to r_b in the investigated pressure range (2 – 13 MPa). The highest pressure exponent ($n = 0.52$) was reached with the GAP-based propellant showing the lowest oxidizer/binder ratio (1.33).

Figure 6 is showing an overview of all measured burning rates in dependence of pressure in the range of 2 MPa – 13 MPa. As mentioned before, the main factor to influence r_b is the choice of the binder. The HTPB samples with AP as oxidizer showing the lowest burning rates in dependence of pressure (HAP_27_73; HAP_21_79). Replacing HTPB with GAP under the condition of keeping AP as oxidizer, r_b is nearly doubled independent of the oxidizer/binder ratio (GAP_27_73; GAP_43_57). The first batch, ADN-based propellants in combination with both binders, shows invariably a higher burning rate compared to the AP-based systems. With the exception of the HTPB/ADN samples, the pressure exponent is spreading between 0.41 and 0.52 independent of the used components and oxidizer/binder ratios. Non-energetic binder loaded with ADN as oxidizer show the best marked pressure sensitivity over the investigated pressure range. With 0.74 (HADN_21_79) and 0.76 (HADN_27_73), it is more the 40 % higher compared to the other formulations.

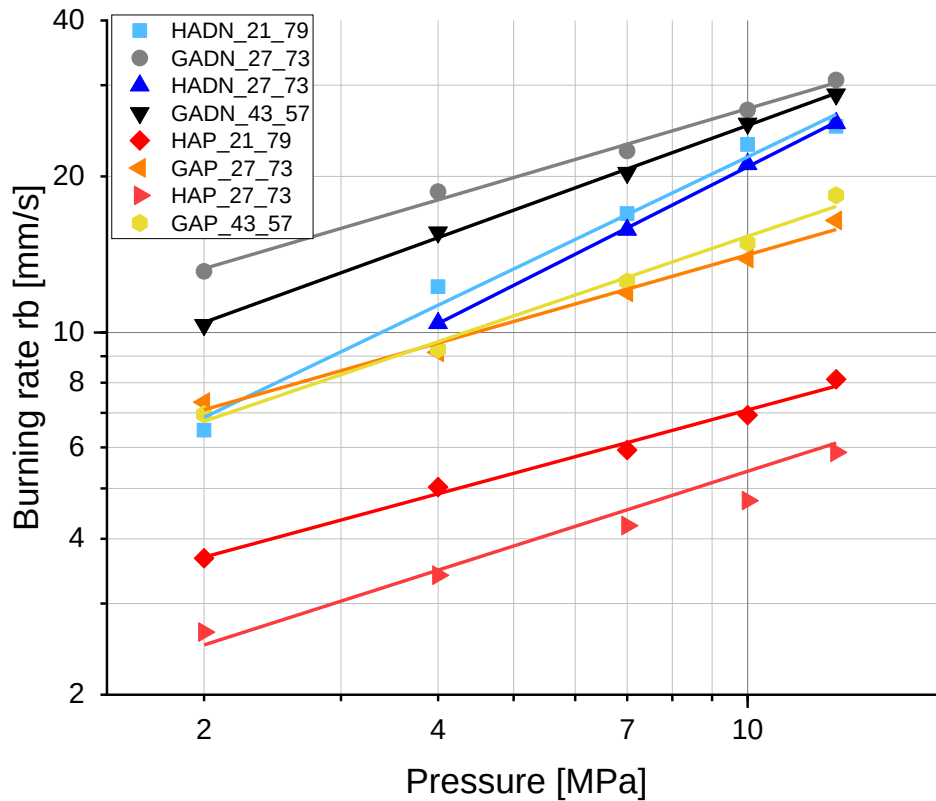


Figure 6 Overview of the calculated burning rate (Vieille's law) in dependence of pressure.

The final step is a comparison of the measured gas phase temperatures at the investigated pressure range (Figure 7). With regard to the calculated adiabatic temperatures (ICT Thermodynamic Code), the measured temperatures are generally lower (Table 7). The explanation is the non-adiabatic experimental set up (window bomb) leading to heat losses to the environment. Nevertheless, the measured flame temperatures reach nearly 75 % of the calculated adiabatic temperatures and the different propellant systems are responsive to the calculated predictions.

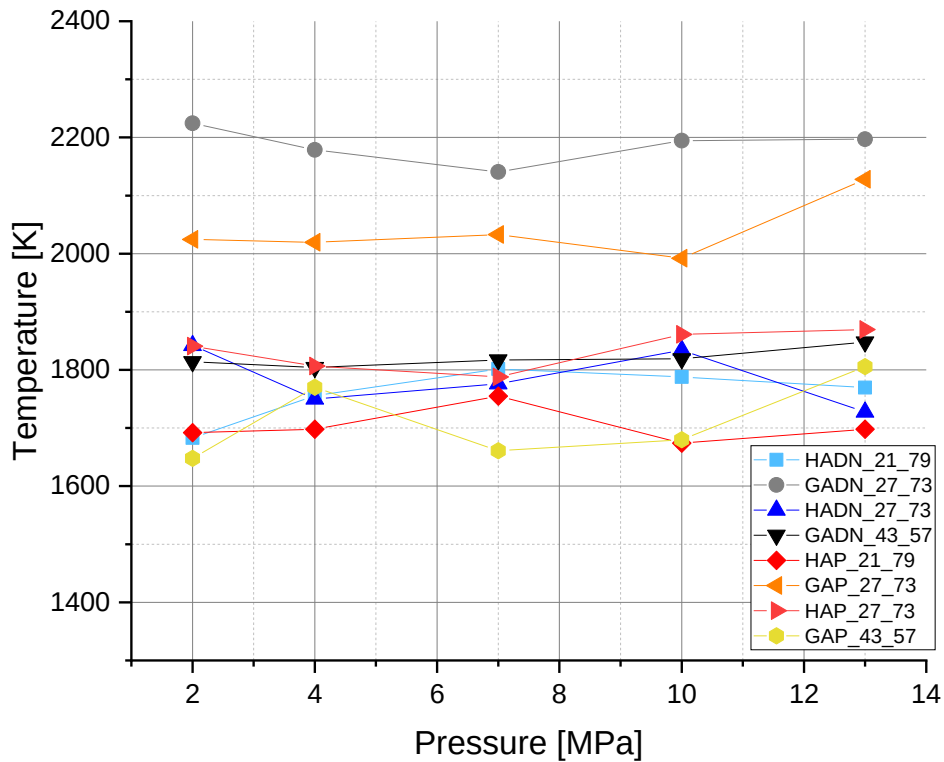


Figure 7 Overview of the measured continuum temperatures of the investigated propellants.

Over the investigated pressure range from 2 MPa – 13 MPa, the measured temperatures are not changing dramatically in dependence of pressure. The main influence factor to the combustion temperature is a combination of two conditions. The choice of an energetic binder (GAP) and a high oxidizer/binder ratio results in high measured flame temperatures. GAP/ADN and GAP/AP propellants with an oxidizer/binder ratio of 2.70 exhibit measured flame temperatures of 2187 K and 2040 K.

The type of the oxidizer (AP or ADN) is not playing a major role. GAP-based compositions with lower oxidizer/binder ratios than 2.7 show measured temperatures similar to AP or ADN in combination with the non-energetic binder HTPB.

Sample	T_{ad} [K]	T_m [K]
HADN_21_79	2115	1760
GADN_27_73	2943	2187
HADN_27_73	2265	1786
GADN_43_57	2265	1820
HAP_21_79	2235	1703
GAP_27_73	3037	2040
HAP_27_73	2332	1833
GAP_43_57	2363	1712

Table 7 Comparison of the calculated (T_{ad}) and measured (T_m) temperatures.

Particularly noticeable, after the investigation of the propellants, is the significant increase of the pressure exponent after replacing the energetic binder (GAP) with a non-energetic one (HTPB) in an ADN-based system. In order to get a better understanding of the combustion mechanism, HTPB/ADN and GAP/ADN samples were quenched in liquid nitrogen near their PDL in the window bomb. The results of the Nano-CT measurement are shown in Figure 8. Because of the density difference between binder and oxidizer, the original photos could be processed and coloured in dependence of the material.

GAP/ADN propellants show steady reacted burning surface, which means that the decomposition process for the binder (GAP) and the oxidizer (ADN) is initiated at similar time and temperature conditions. This is a basic requirement for an effective burning of a propellant. In comparison to this burning behaviour, HTPB/ADN formulations create “oxidizer pockets” near to the burning surface. This fact implies a non-steady reaction of binder and oxidizer. ADN is decomposing earlier at lower temperature conditions than the non-energetic binder HTPB. An optimal gas-phase-reaction of the gaseous binder and oxidizer products is not given.

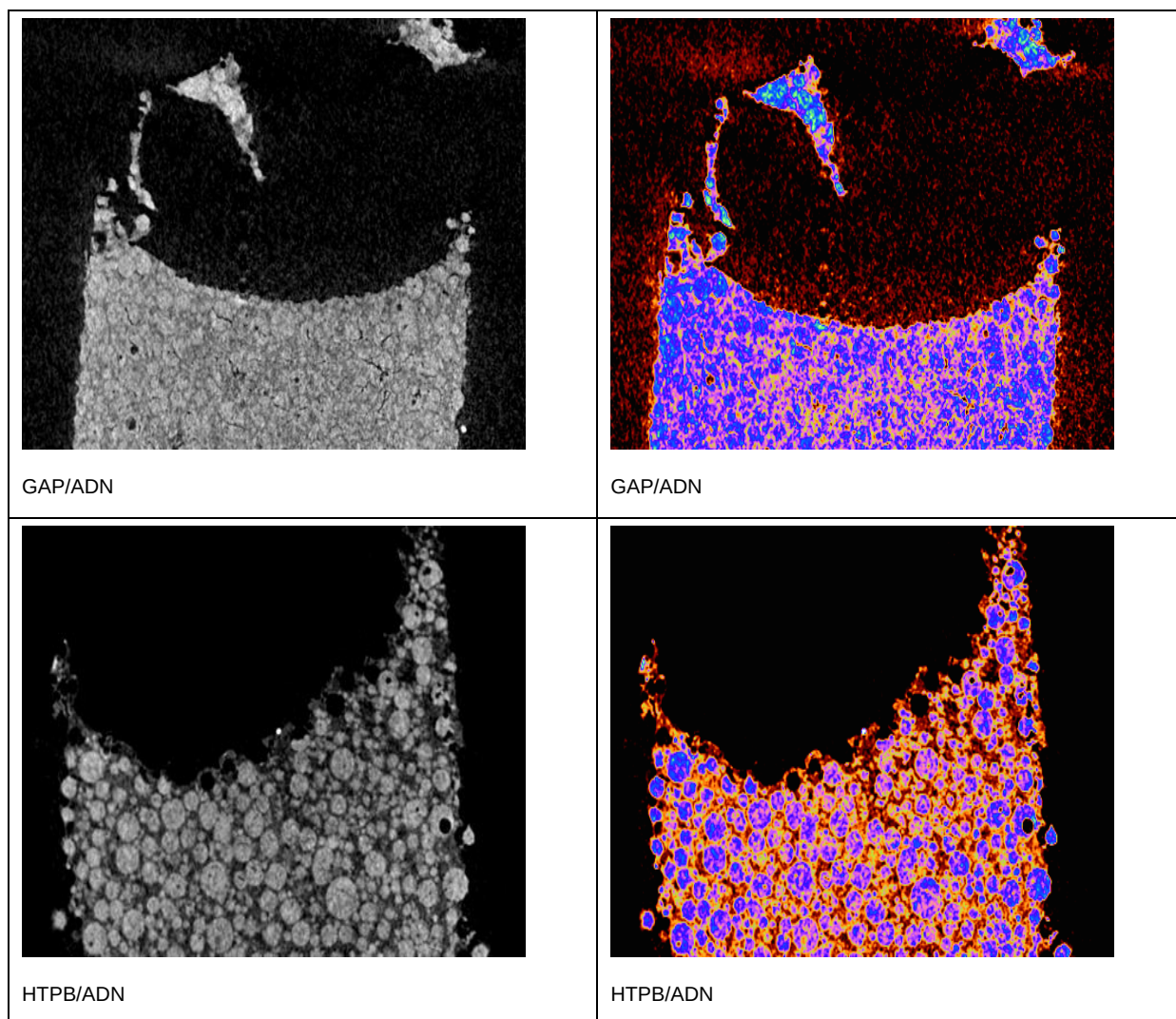


Figure 8 Nano-CT images of GAP/ADN and HTPB/ADN propellants: original photo (left); coloured photo (right) in dependence of component (yellow/red: binder; purple/blue: oxidizer).

In addition to the Nano-CT analysis, Raman-mapping of the quenched GAP/ADN and HTPB/ADN samples was performed with the aim to get information about the quenched reaction products of the combustion near the surface and about possible differences of the reactions process in dependence of the location in the solid phase (X-direction; Figure 9).

In a first step, Raman spectra of the single components for both systems were taken. In dependence of the analysed system, HTPB, GAP and ADN spectra form the base for the following mapping. The measurements were accomplished with a Raman confocal microscope from Horiba. Based on the information of the single component spectra, a 633 nm laser has been used and the spectral measurement range was set to $200 - 2000 \text{ cm}^{-1}$. The region of interest (ROI) for the mapping was set to $600 \mu\text{m} \times$

600 μm (three times the mean particle size of the ADN prills). In summary, the ROI contains 400 individual measurement points with a distance of 30 μm (Figure 9; left).

The target spectral array for both systems (GAP/ADN, HTPB/ADN) is least squares fitted with the selected (pure ingredients) spectra. Such a method is ideally suited for the identification of each spectrum in the mapping. In summary, three single components could be located for both investigated formulations (Figure 9, right): unreacted binder (GAP/HTPB; red), unreacted oxidizer (ADN; blue) and soot (reacted binder; grey).

For the GAP/ADN investigated formulation, the soot dominated layer reveals a width of 176 μm (Figure 9; GAP/ADN, right). This is exactly the average particle size of the used ADN prills. Also, one unreacted ADN particle could be identified in the soot layer. From 0 – 420 μm (X-direction) and over the whole Y-range, no soot could be found anymore and the distribution of binder and oxidizer is completely homogeneous as in the initial sample. This fact is approving the steady reacted burning surface, based on the evaluation of the Nano-CT analysis.

A completely different result could be seen in the Raman-mapping of the HTPB/ADN sample (Figure 9; HTPB/ADN, right). Compared to the GAP/ADN formulation, the soot layer width of reacted binder is doubled. The “oxidizer pockets” are located in the soot layer between 320 μm and 580 μm in X-direction (red area) and also in the oxidizer/binder layer in the range of 50 μm – 180 μm . The pockets in the soot layer and the soot traces in the oxidizer/binder layer indicate a premature decomposition of the ADN particles towards the used non-energetic binder HTPB.

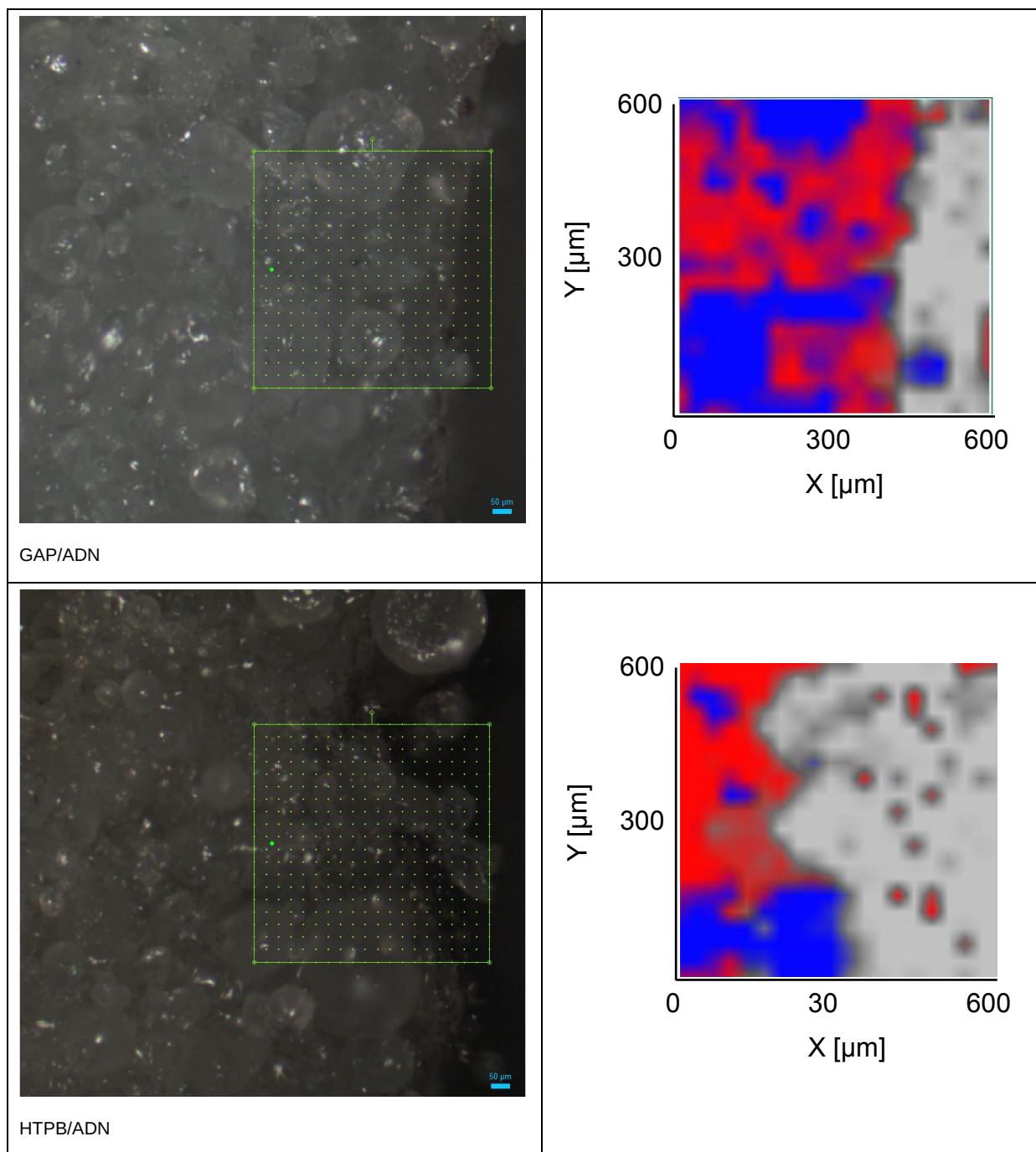


Figure 9 Raman-mapping of GAP/ADN and HTPB/ADN propellants. Left: Overview of the region of interest; right: Mapping results, coloured in dependence of the component (binder (red); oxidizer (blue); soot (grey)).

4 Summary

The combustion behavior of GAP/ADN, HTPB/ADN and GAP/AP, HTPB/AP propellants was studied. Samples were tested in a window bomb at 2, 4, 7, 10 and 13 MPa nitrogen pressure. A fundamental difference in the combustion behavior between ADN and AP based propellants could be attested. The significant difference

of the pressure exponent between GAP/ADN and HTPB/ADN propellants is attributed to the premature decomposition of the oxidizer compared to the binder and the resulting ineffective combustion.

5 Abbreviation

ADN	Ammonium dinitramide
AP	Ammonium perchlorate
GAP	Glycidyl azide polymer
HTPB	Hydroxyl-terminated polybutadiene
Al	Aluminium
Mg	Magnesium
HMX	High-Molecular-weight RDX
RDX	Royal Demolition Explosive
Cat.	Catalyst
ICT	Institut für Chemische Technologie
ICT Code	ICT Thermodynamic code from Fraunhofer ICT
I_{sp}	Specific impulse
n	Pressure exponent
OX	Oxidizer
PB	Polymeric binder
r_b	Burning rate
EUB	EURENCO Bofors
PDL	Pressure Deflagration Limit
ROI	Region of interest

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