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RESEARCH ARTICLE OPEN ACCESS

Comparative Assessment of Energetic Plasticizers Including NFPEG3N3 in a GAP/HMX Formulation: Mechanics, Stability, Combustion, and Performance

Patrick Lieber[1] | Uwe Schaller[1] | Thomas M. Klapötke[2]

[1]Fraunhofer Institute for Chemical Technology, Pfinztal, Germany | [2]Department of Chemistry, Ludwig-Maximilian-University of Munich, Munich, Germany

Correspondence: Patrick Lieber (patrick.lieber@ict.fraunhofer.de)

Received: 10 October 2025 | **Revised:** 11 November 2025 | **Accepted:** 14 November 2025

Keywords:1,2,5-oxadiazole | energetic plasticizer | glycidyl azide polymer | heat-flow microcalorimetry | Vieille's law

ABSTRACT

We examine the new energetic plasticizer 3-(2-(2-(2-azidoethoxy)ethoxy)ethoxy)-4-nitro-1,2,5-oxadiazole (NFPEG3N3), three reference compounds (BDNPA/F, Bu-NENA, and DNDA-57), and a plasticizer-free control in a fixed HMX/GAP composite. NFPEG3N3 provides the strongest low-temperature properties, yielding the lowest formulation glass transition temperature of −50.9°C. In tensile testing, NFPEG3N3, Bu-NENA, and DNDA-57 show comparable peak strengths (0.36–0.38 MPa), while BDNPA/F is higher (0.50 MPa). NFPEG3N3 achieves the highest ductility (3.8% strain at maximum stress) and the lowest elastic modulus (16.7 MPa), indicating superior compliance without a penalty in peak load capacity relative to the other energetic plasticizers. All formulations meet STANAG-referenced stability criteria in vacuum stability, heat-flow microcalorimetry, and mass loss. Consistent with its nitrate ester-free structure, NFPEG3N3 exhibits distinctly lower heat release and mass loss than BDNPA/F and Bu-NENA. Burning-rate regression shows NFPEG3N3 does not significantly change the burn rate at 7 MPa, while reducing the Vieille pressure exponent by 16.5% to 0.792. As polymer-bonded explosives, DNDA-57 and BDNPA/F yield the highest detonation velocity and pressure, while NFPEG3N3 is slightly above the control in detonation velocity and notably higher in pressure and temperature of detonation. As composite propellants, NFPEG3N3 matches Bu-NENA in mass-specific impulse and exceeds it in volume-specific impulse. Overall, NFPEG3N3 has balanced properties: enhanced toughness, acceptable strength, favorable stability, and improved combustion characteristics. These properties make it a promising alternative to nitrate ester plasticizers in GAP/HMX systems.

1 | Introduction

Polymer-bonded energetic materials pair crystalline oxidizers, metallic fuels, or explosives with a polymeric binder to balance energy, processability, and mechanical integrity [1–3]. Glycidyl azide polymer (GAP) is a widely used energetic binder whose high nitrogen content and positive heat of formation can enhance performance relative to inert matrices such as hydroxyl-terminated polybutadiene (HTPB) [4]. A well-known limitation, however, is GAP's brittleness, especially at low temperatures, which promotes microcracking, debonding, and defect growth under thermal or mechanical loads [5]. Achieving a sufficiently low glass transition temperature (T_g) and adequate strain capacity is therefore essential to preserve structural integrity and predictable ballistic or detonation behavior.

Plasticizers are routinely employed to tune the viscoelastic response of cured GAP networks by increasing chain mobility and lowering T_g, thereby improving ductility [6, 7]. Plasticizers can contribute to energy release, oxygen balance, and combustion

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https://doi.org/10.1002/prep.70095

characteristics when they are energetic. Their selection entails trade-offs spanning curing behavior, filler-binder interactions, thermal stability and aging, sensitivity, and effects on burning rate and pressure dependence. Although legacy energetic plasticizers such as BDNPA/F [7, 8], Bu-NENA [9], and DNDA-57 [10] have extensive histories of research, nitrate ester functionalities can raise concerns about thermally driven aging and compatibility margins in certain systems. This has motivated interest in nitrate-ester-free energetic plasticizers that provide robust mechanical compliance and storage stability without sacrificing performance [11].

3-(2-(2-(2-Azidoethoxy)ethoxy)ethoxy)-4-nitro-1,2,5-oxadiazole (NFPEG3N3) is a novel energetic plasticizer featuring a nitrofurazanyl and an azide moiety [12]. It has demonstrated compatibility with GAP diol, isocyanate, and Octogen (HMX), suggesting chemical stability in GAP/HMX composites [13]. Despite the central role of plasticizers in GAP-based systems, there are limited comprehensive, side-by-side comparisons that integrate mechanical, thermal, stability, and combustion metrics within a single, fixed formulation. To our knowledge, NFPEG3N3 has not been benchmarked against other energetic plasticizers using this framework.

2 | Experimental Section

2.1 | Safety Statement

All work with energetic materials (HMX, GAP, BDNPA/F, Bu-NENA, DNDA-57, NFPEG3N3) was performed by trained personnel in facilities authorized for explosives handling. Some of the procedures described in this publication, particularly those involving the mixing and curing of formulations, require the use of protective equipment and remote control.

2.2 | Materials

Desmodur N100 was purchased from Covestro Deutschland AG, Germany. GAP diol (charge 06S15, MW 1814 g/mol, EQ 1228 g/mol, functionality 1.5) was purchased from Eurenco, France. HMX was supplied by Eurenco Bofors AB, Sweden. Dibutyltin dilaurate (D22) was purchased from Sigma-Aldrich. Bu-NENA and BDNPA/F were purchased from Chemring, Sweden. DNDA-57 was obtained from the N. D. Zelinsky Institute of Organic Chemistry, Russia.

2.3 | Synthesis

NFPEG3N3 was synthesized at Fraunhofer ICT according to the literature [12].

2.4 | Composition

Table 1 shows the detailed composition of the plasticized formulations investigated in this study: NFPEG3N3 (GHX213), BDNPA/F (GHX193), Bu-NENA (GHX196), and DNDA-57 (GHX197), as well as the control formulation without plasticizer (GHX192). After curing, the mass of GAP is considered as the sum of GAP diol and Desmodur N100.

TABLE 1 | Composition of plasticized and non-plasticized formulations.

Compound	GHX193, 196, 197, 213 mass (wt.%)	GHX192 mass (wt.%)
HMX Grade B Class 3	53.90	53.90
HMX Grade B Class 5	23.10	23.10
Gap diol 06S15	15.96	19.95
Energetic plasticizer	4.60	—
Desmodur N100	2.44	3.05
D22 (Catalyst)	0.000575	0.000345

2.5 | Processing

A total of 200 g of all compounds was weighed and mixed in a Resodyne (Butte, MT 59701, USA) LabRAM acoustic mixer at a g-force of 70 g for 750 s in a 250-mL vessel. Then, the mixture was degassed at 15 mbar for 180 s. During the mixing process, the temperature of the material increased from 22.7°C to 45.1°C. The mixture was then poured into molds on a vibrating table. The material was then cured for 7 days at 50°C.

2.6 | Instruments and Methods

Glass transition temperature was determined using a TA Instruments Q1000 differential scanning calorimeter (DSC) and perforated aluminum crucibles. The sample was first cooled to −90°C and then heated at a rate of 10°C/min under a nitrogen flow of 25 mL/min. The glass transition temperature was taken as the inflection point of the heat flow curve during the heating cycle.

Mechanical properties of the cured formulations were determined with a Zwick-Roell UPM 1476 tensile test machine. The mini-dog bone specimens [14] were punched from plates and stored over silica gel desiccant. The measurements were performed at 20°C to 23°C and atmospheric pressure in uniaxial tensile mode with a crosshead speed of 50 mm/min. The effective gauge length of the specimen was 16 mm. The following numbers of specimens could be tested: Thirteen specimens for NFPEG3N3, six for the unplasticized control, six for BDNPA/F, six for Bu-NENA, and seven for DNDA-57. The difference in the number of specimens is due to losses caused by damage during sample preparation.

Burning rates were measured under a nitrogen atmosphere. Test samples, in the form of strands measuring 5 × 5 × 120 mm^3, were cut from plates. These samples were burned in the optical bomb, a high-pressure autoclave equipped with glass windows, allowing for non-intrusive combustion measurements. A detailed outline of the approach is published elsewhere [15–18]. In this study, a color high-speed camera (Redlake MotionPro X3) and an emission spectrometer (ZEISS MCS 611 NIR 2.2) were employed. The ignition was carried out using ignition paste. Burning rates were derived from video recordings through image post-processing. A total of nine measurements were performed for the formulation with NFPEG3N3 (three measurements at 3 MPa, one measurement at 5 and 13 MPa, and two measurements each at 7 and 10 MPa). A total of 10 measurements were taken for each reference formulation (two measurements each at 3, 5, 7, 10, and 13 MPa).

Heat-flow microcalorimetry (HFMC) was performed using a TAM (Thermal Activity Monitor) Type III from TA Instruments (subsidiary of Waters Corporation, USA). The instrument was originally developed by Thermometric AB, Sweden. Samples of 1 g were filled into a glass vial, which was placed inside an air-filled 4 mL stainless-steel ampoule. Testing under air conditions is specified in STANAG 4147 to simulate realistic conditions. It introduces oxygen into the test environment, which, for example, can influence oxidation or corrosion reactions between the explosive and the metal. The steel ampoule was closed tightly and placed into the measuring device, which was submerged in an oil bath under isothermal conditions. The measurements were conducted at 90°C for a 20-day test period.

Vacuum stability test (VST) was estimated by glass sample tubes that were filled with 2.5 g of the test material and connected to a mercury-filled manometer. The mercury served both as a measuring medium and as a seal for the apparatus. The sample tube and the manometer were evacuated, and the ambient temperature and atmospheric pressure were recorded. The sample was then heated to 100°C and maintained at this temperature for 40 h. After the heating period, the system was allowed to cool to room temperature. The volume of gas released during the test was determined by measuring the displacement of the mercury column in the manometer. Final ambient temperature and pressure were again recorded. Based on the measured gas volumes and the recorded environmental conditions, the gas volumes were corrected to standard temperature (0°C) and pressure (1 atm).

Mass loss (ML) samples of 2 g each were stored in glass vials with a diameter of 17 mm (two replicates per formulation). The vials were closed with loosely inserted ground stoppers that were not greased or clamped. The samples were stored in PID-controlled aluminum block ovens. Vial masses were recorded using an analytical balance with a precision of 0.1 mg.

Propulsion performance of the formulations as composite propellants was evaluated using the ICT Thermodynamic Code V2017.1.1 [19] under idealized conditions with a nozzle expansion ratio of 70:1 in the frozen equilibrium mode. During the calculations, water was treated as gaseous. Unless otherwise specified, the thermochemical properties of the components were obtained from the ICT database.

Detonation performance of the formulations as PBX was evaluated using Explo5 V6.06 [20, 21] and the BKW EOS at an initial guess temperature of 3600°C and an initial pressure of 0.1 MPa.

Mechanical sensitivity was estimated with a BAM drop hammer and BAM friction device, in accordance with DIN EN 13631.

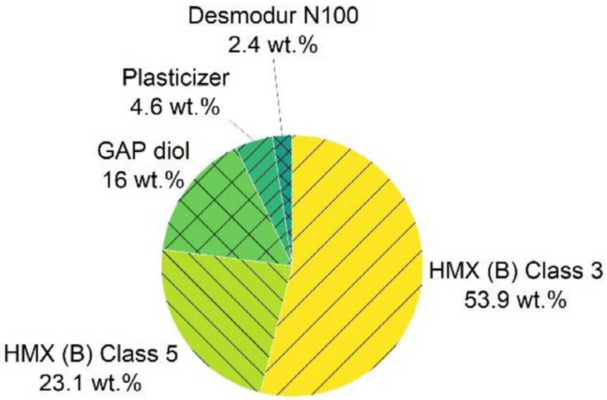

FIGURE 1 | Composition of plasticized formulations investigated.

3 | Results and Discussion

3.1 | Composition and Processing

To investigate the impact of NFPEG3N3 on an actual energetic formulation for the first time, a GAP-based formulation containing 77 wt.% HMX filler and 23 wt.% binder was selected. The HMX consisted of a bimodal mixture of 53.9 wt.% Grade B Class 3 and 23.1 wt.% Grade B Class 5. The binder cures to a polyurethane network through the reaction of GAP diol with the trifunctional isocyanate Desmodur N100. The binder comprised 18.4 wt.% GAP and 4.6 wt.% plasticizer, with the plasticizer accounting for 20% of the binder content by weight. The composition of the plasticized formulations is shown graphically in Figure 1. This formulation could serve as a weaker explosive charge or as a sustainer stage in a solid rocket motor. It was chosen primarily for its robustness and exemplary nature. In addition to the new nitrofurazanyl plasticizer NFPEG3N3, the well-known energetic plasticizers Bu-NENA, DNDA-57, and BDNPA/F were used to create the reference formulations. A control formulation without plasticizer was also prepared, in which the plasticizer fraction was replaced by GAP. All formulations were prepared by acoustic mixing and cured at 50°C. The cured NFPEG3N3 formulation had an impact sensitivity of 15 J and a friction sensitivity of 324 N, enabling the new material to be stamped, cut, or otherwise processed to produce the necessary test specimens for the tensile tests shown in Figure 2.

3.2 | Mechanical Properties

3.2.1 | Glass Transition Temperature

The glass transition temperature (T_g) of the formulation with NFPEG3N3 was measured by DSC to −50.9°C. The control without plasticizer exhibited a T_g of −36.8°C. References containing DNDA-57, BDNPA/F, and Bu-NENA showed T_g values of −39.7°C, −40.3°C, and −49.3°C, respectively. Among the tested plasticizers, the formulation with NFPEG3N3 exhibited the lowest T_g, indicating a superior low-temperature plasticization capability of the novel compound. Figure 3 shows a comparison of the measured T_g.

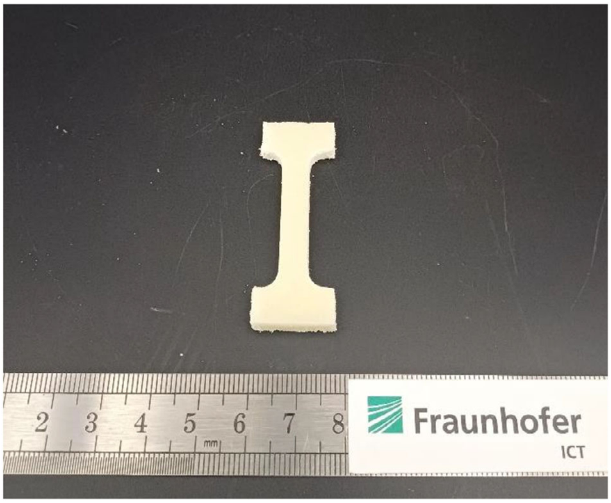

FIGURE 2 | Dog bone specimen of the NFPEG3N3 plasticized formulation.

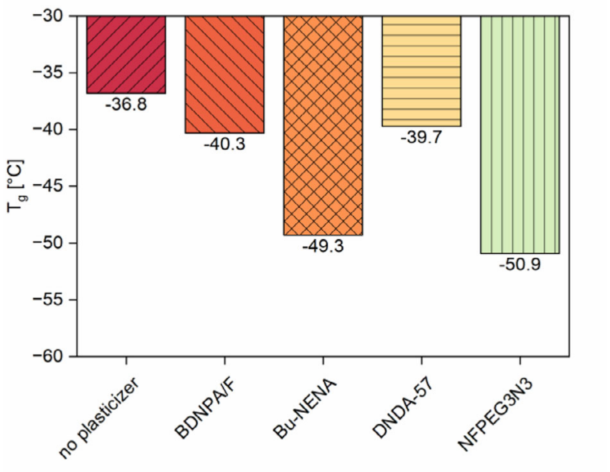

FIGURE 3 | Glass transition temperatures of the investigated formulations.

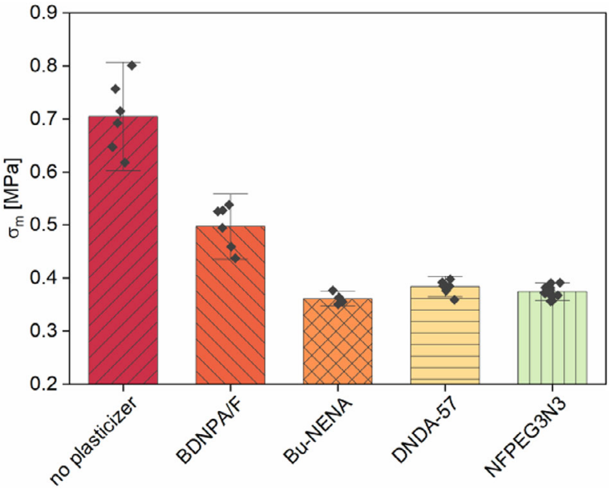

FIGURE 4 | Maximum stress.

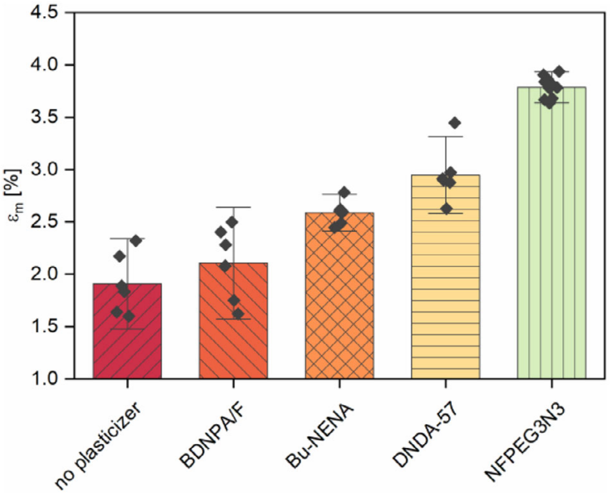

FIGURE 5 | Strain at maximum stress.

3.2.2 | Tensile Test

The mechanical properties of the formulations were evaluated using a tensile testing machine. As expected, the presence of plasticizers led to a reduction in maximum stress compared to the unplasticized formulation (Figure 4). The formulations containing NFPEG3N3, Bu-NENA, and DNDA-57 exhibited similar maximum stress values, ranging from 0.36 to 0.38 MPa. In contrast, the BDNPA/F-containing formulation showed a higher value of 0.50 MPa, indicating it can withstand a greater load before undergoing plastic deformation. All stress-strain curves and detailed data have been added to the Supporting Information. The strain at maximum stress provides insight into the ductility of a material (Figure 5). A higher value indicates that the material can elongate more before reaching its peak strength. The increase in this parameter reflects the effectiveness of the plasticizer. Among the plasticizers tested, NFPEG3N3 exhibited the strongest plasticizing effect, with an elongation at maximum stress of 3.8%, followed by DNDA-57 (2.9%), Bu-NENA (2.6%), and BDNPA/F (2.1%). The unplasticized sample showed the lowest elongation at only 1.9%. NFPEG3N3 thus demonstrates superior plasticizing performance, enabling greater elongation at comparable maximum stress. Figure 6 shows a plot of these two properties against each other. This is further supported by the formulation's lowest elastic (Young's) modulus of 16.7 MPa, indicating to be the overall most flexible material in the study (Figure 7).

3.3 | Stability

We evaluated the stability of the formulation with NFPEG3N3, as well as references with BDNPA/F and Bu-NENA, and a control without plasticizer, using VST, HFMC, and ML testing. VST was performed according to STANAG 4556 Ed. 2 [22]. A gas generation limit of 1.2 mL/g over 40 h at 100°C was applied, as specified in TL 1376-800 by the German military. HFMC was performed according to STANAG 4582 Ed. 1 [23] with a heat generation limit of 103.8 J/g and a heat flow limit of 350 µW/g over 3.43 days, respectively [24]. For ML, the frequently cited limit of 3% over 18 days was used as the basis [25]. It originates from the German military's technical delivery conditions for gun propellants. For

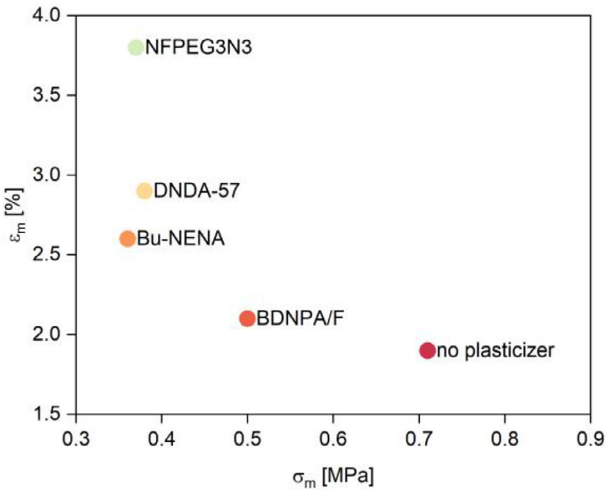

FIGURE 6 | Maximum stress against strain at maximum stress.

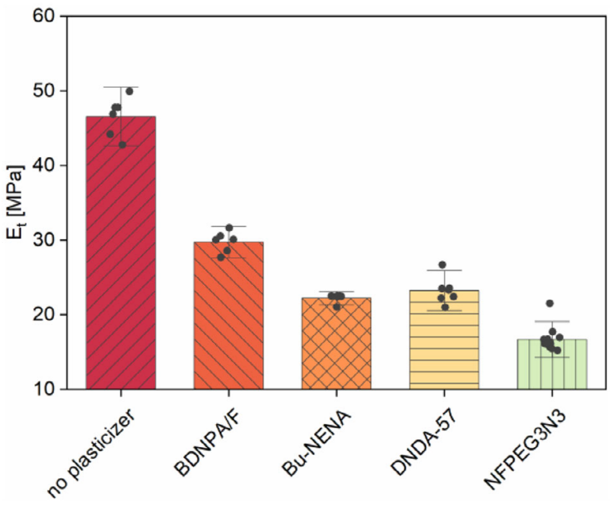

FIGURE 7 | Elastic (Young's) modulus.

the longer investigation period of 21 days in this study, it was extrapolated linearly to 3.5%. An extended measurement period helps to detect slow or delayed gas-forming reactions. As shown in Table 2, all formulations meet the stability requirements in the respective tests. The formulation without plasticizers shows the best stability values, as GAP has been replaced by more energy-rich but also more unstable components in the other formulations. In HFMC and ML, NFPEG3N3 performs significantly better than the comparative plasticizers. One advantage could be that, unlike the reference substances, NFPEG3N3 does not contain any aliphatic nitro or nitrate ester groups. In the case of VST, the new formulation is in the area of comparative plasticizers.

The heat generation and heat flow curves are shown in Figure 8. HFMC measurements were extended to 20 days to ensure the detection of slow or delayed exothermic reactions. The curve for the formulation with Bu-NENA shows a sharp increase at the beginning and flattens out significantly after 1 day. It is possible that decomposition products of the nitrate ester compound have already accumulated during the 7-day curing period at 50°C, which react at the start of the measurement at 90°C. In all cases, the heat flow is nearly constant and without abnormalities after a short period of time. Figure 9, showing the ML over 21 days, also reveals the significantly steeper increase in the Bu-NENA-containing formulation.

The results indicate that GAP-based formulations containing NFPEG3N3 and HMX meet the standard thermal stability criteria and are highly likely to be suitable for long-term storage. In addition, the formulation investigated in this study demonstrates higher thermal stability than the reference samples containing Bu-NENA and BDNPA/F in HFMC and ML investigations.

3.4 | Performance Evaluation

3.4.1 | Burning Rate

Burning-rate measurements were carried out on the formulation with NFPEG3N3 and three reference formulations. The burning-rate behavior at a pressure range from 3 to 13 MPa was characterized by the empirical Vieille's law (Equation 1), where n is the dimensionless pressure exponent, r is the burn rate, p is the pressure, and a is a pre-exponential coefficient. Results are presented in Table 3.

Equation 1: Vieille's burning-rate law.

$$r = a \cdot p^n$$

TABLE 2 | Summary of stability results and limits.

	VST[a]	HFMC[b]		ML[c]
T/t	100°C/40 h	90°C/3.43 d		90°C/21 d
Limit	$V < 1.2$ mL/g	$Q < 103.8$ J/g	Max $dQ/dt < 350$ μW/g	ML < 3.5%
No plasticizer	0.330	2.04	20	0.13
BDNPA/F	0.450	5.95	140	0.17
Bu-NENA	0.780	20.39	179	0.87
NFPEG3N3	0.630	4.81	112	0.16

[a] Vacuum stability test.

[b] Heat-flow microcalorimetry.

[c] Mass loss.

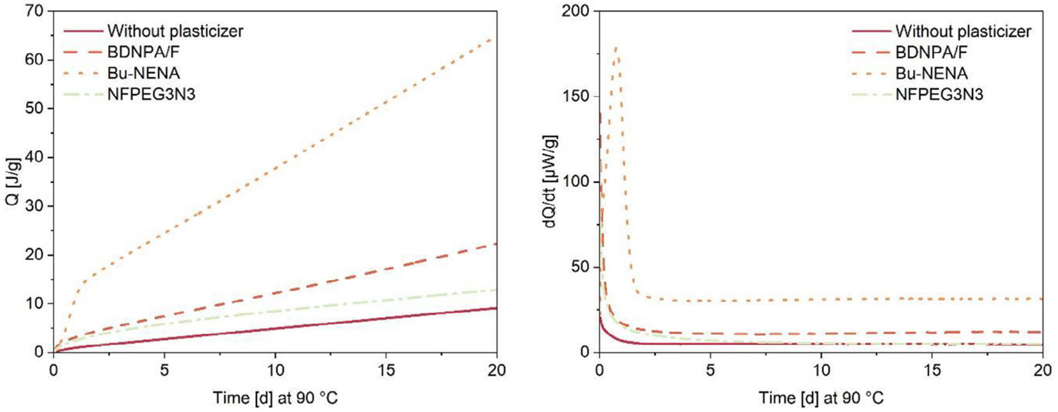

FIGURE 8 | Heat generation (left) and heat flow curves (right) of investigated formulations.

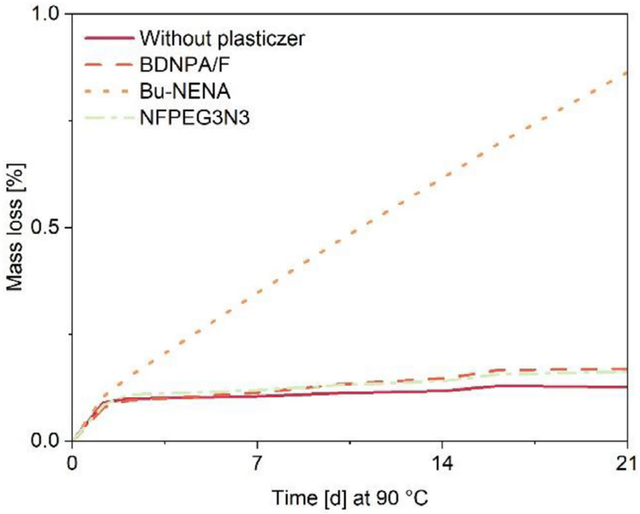

FIGURE 9 | Mass loss curves of investigated formulations.

NFPEG3N3 reduced the pressure exponent by 16.5% relative to the control, a larger reduction than BDNPA/F (9%). Bu-NENA has no significant effect on the pressure exponent. BDNPA/F and Bu-NENA reduce the burn rate at 7 MPa, whereas NFPEG3N3 does not cause a significant change. A lower pressure exponent positively impacts stability, robustness, and predictability of performance by making the propellant less sensitive to pressure fluctuations.

3.4.2 | Polymer-Bonded Explosive

Detonation performance of the GAP/HMX formulations was computed using Explo5 V6.06. The calculated parameters are summarized in Table 4. In general, energetic plasticization raises detonation pressure and temperature relative to the inert plasticizers di-octyl adipate (DOA) and the plasticizer-free control. It also shifts the oxygen balance towards neutrality. Among the energetic set, DNDA-57 and BDNPA/F have the highest detonation velocity (8330–8324 m/s) and detonation pressure (approximately 28.13 GPa). This is consistent with their favorable oxygen balance and energy content. NFPEG3N3 yields detonation performance above DOA and the control in detonation pressure (+ 0.37 GPa vs. control) and temperature of detonation (+ 19°C vs. control), with detonation velocity slightly above the control (8293 vs. 8284 m/s). Bu-NENA is close to the control in detonation velocity and pressure. The magnitude of the heat of detonation Q_{CJ} increases for all energetic plasticizers relative to DOA and the control, with BDNPA/F showing the largest value. Collectively, these results indicate that NFPEG3N3 delivers appropriate PBX detonation performance while complementing the mechanical and stability advantages demonstrated elsewhere in this work.

3.4.3 | Solid Rocket Propellant

We calculated the performance of the formulation as a solid rocket propellant using the ICT Thermodynamic Code V2017.1.1. The results are displayed in Table 5. Compared

TABLE 3 | Results of burning-rate regression by Vieille's law.

Formulation	r[a] (mm/s) at 7 MPa	n[b]	a[c] (mm/s)	R^2 [d]
No plasticizer	9.56 ± 1.53	0.948 ± 0.031	0.141 ± 0.020	0.994
BDNPA/F	7.09 ± 0.77	0.859 ± 0.017	0.185 ± 0.015	0.998
Bu-NENA	7.27 ± 1.55	0.940 ± 0.034	0.134 ± 0.021	0.993
NFPEG3N3	9.24 ± 3.12	0.792 ± 0.055	0.319 ± 0.078	0.976

[a] Burn rate from the estimated Vieille parameters at a given pressure.

[b] Pressure exponent.

[c] Pre-exponential coefficient.

[d] Coefficient of determination (COD).

TABLE 4 | Detonation performance parameters calculated by Explo5 (PBX composition: 77 wt.% HMX, 18.4 wt.% GAP, 4.6 wt.% plasticizer).

Plasticizer	ρ[a] (g/cm^3)	D[b] (m/s)	P[c] (GPa)	T[d] (°C)	Q_{CJ}[e] (kJ/Kg)	OB_{CO2}[f] (%)
DOA	1.68	8037	25.36	3276	−4999	−51.0
without	1.72	8284	27.42	3394	−5180	−44.5
BDNPA/F	1.73	8324	28.14	3438	−5247	−41.6
Bu-NENA	1.71	8268	27.49	3405	−5207	−43.7
DNDA-57	1.72	8330	28.13	3422	−5229	−42.3
NFPEG3N3	1.72	8293	27.79	3413	−5195	−43.0

[a] Theoretical maximum density (TMD).
[b] Detonation velocity.
[c] Detonation pressure.
[d] Detonation temperature.
[e] Heat of detonation.
[f] Oxygen balance assigned to CO_2.

TABLE 5 | Propulsion performance parameters calculated by ICT thermodynamic code (propellant composition: 77 wt.% HMX, 18.4 wt.% GAP, 4.6 wt.% plasticizer).

Plasticizer	ρ[a] (g/cm^3)	I_{sp}[b] (s)	V_{sp}[c] (N s/dm^3)	T_c[d] (°C)	T_a[e] (°C)
DOA	1.68	232.8	3835	2325	851
without	1.72	241.5	4075	2578	975
BDNPA/F	1.73	244.2	4138	2667	1028
Bu-NENA	1.71	243.1	4087	2610	995
DNDA-57	1.72	244.4	4134	2654	1018
NFPEG3N3	1.72	243.0	4110	2630	1004

[a] Theoretical maximum density (TMD).
[b] Mass-specific impulse.
[c] Volume-specific impulse.
[d] Chamber temperature.
[e] Nozzle temperature.

to a formulation without or with an inert plasticizer, NFPEG3N3 increases the mass and volume-specific impulse. Among the energetic plasticizers, mass-specific impulse ranks DNDA-57 ≈ BDNPA/F > Bu-NENA ≈ NFPEG3N3. Volume-specific impulse ranks BDNPA/F ≈ DNDA-57 > NFPEG3N3 > Bu-NENA. Accordingly, NFPEG3N3 matches Bu-NENA in mass-specific impulse, while in volume-specific impulse it clearly surpasses Bu-NENA but remains below BDNPA/F and DNDA-57. Examining the calculated reaction products revealed that none of the evaluated formulations produced condensed reaction products, particularly solid carbon.

4 | Conclusion

Among the plasticizers investigated, NFPEG3N3 delivered the strongest overall plasticization. It produced the lowest glass transition temperature (−50.9°C) and the highest ductility (3.8% strain at maximum stress) at comparable strength to Bu-NENA and DNDA-57. These results indicate superior low-temperature compliance and reduced brittleness relative to other energetic plasticizers. As expected, the plasticizer-free control was the most stable. However, NFPEG3N3 showed distinctly lower heat release and ML than BDNPA/F and Bu-NENA. The stability advantages of a plasticizer that does not contain aliphatic nitro or nitrate ester groups are evident. All formulations met the German military standards for gun propellants in terms of VST performance. Combustion testing showed NFPEG3N3 did not significantly change the 7 MPa burn rate versus the plasticizer-free control while reducing the pressure exponent significantly. Relative to BDNPA/F and Bu-NENA, NFPEG3N3 exhibited, with approximately 9 mm/s, a higher 7 MPa burn rate. The reduced pressure sensitivity with maintained burn rate is favorable for propellant robustness and controllability. Thermochemical modeling indicated that NFPEG3N3 is competitive in mass- and volume-specific impulse with Bu-NENA, though DNDA-57 and BDNPA/F retain an advantage in mass-specific impulse. Overall, NFPEG3N3 offers enhanced low-temperature toughness and favorable aging behavior while maintaining or improving combustion characteristics. This positions NFPEG3N3 as a promising alternative when storage stability and low-temperature handling are priorities.

Acknowledgments

We thank A. Happ for formulation processing, G. Kronis for specimen preparation and tensile testing, H. Schuppler for DSC measurements, D. Bieroth and P. E. Pietrek for burn-rate measurements, C. Müller and T. Grunwald for stability tests, and M. Jung for mechanical sensitivity testing. Financial support from WTD91 of the German Ministry of Defense is also gratefully acknowledged.

Open access funding enabled and organized by Projekt DEAL.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data supporting this article, including detailed tensile test results and DSC curves, have been included as part of the Supporting Information.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

Supporting File 1: prep70095-sup-0001-SuppMat.docx