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Nitrogen-Rich Salts as Energetic Additives for Hypergolic Ionic Liquids with Hydrogen Peroxide

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Abstract

For the substitution of toxic and environmentally harmful hypergolic bipropellants, the combination of ionic liquids and highly concentrated hydrogen peroxide is investigated. In this work, guanidinium thiocyanate [Gua][SCN] and choline thiocyanate [Ch][SCN] were tested as energetic additives for hypergolic ionic liquids. Viscosities and densities of the new fuels were measured, heat of formation was determined and specific impulse was calculated. Ignition behavior of the propellants was evaluated via drop-on-pool tests. The ignition delay time – the time span between first contact of oxidizer and fuel and first formation of a flame – of the different hypergolic propellant combinations was compared and the influence of the additives on the ignition capability was investigated. Overall, it was found that [Gua][SCN] was able to reduce the ignition delay compared to the base fluid [Emim][SCN] but did decrease the specific impulse. In case of the additive [Ch][SCN], the reverse was found: here, both a gentle increase in the specific impulse and the volumetric specific impulse could be achieved.

1. Introduction

New green hypergolic propellant combinations are developed by DLR's Institute of Space Propulsion. The development focuses on a suitable replacement of conventional hypergolic propellants like hydrazine, monomethylhydrazine (MMH) or unsymmetrical dimethylhydrazine (UDMH) as fuels with dinitrogen tetroxide (NTO) as oxidizer. These fuels are classified as toxic, carcinogenic and environmentally harmful which makes the usage of these substances hazardous and costly. In 2011, Hydrazine was listed as a "substance of very high concern" in the European REACH (registration, evaluation, authorization of chemicals) regulation, so a prohibition of this substance could be forthcoming. [1]

For the research on alternative hypergolic propellants, high test peroxide (~98 wt%, HTP) was chosen as oxidizer, as it is considered "greenest" compared to other candidates such as NTO, mixed oxides of nitrogen (MON) and white or red fuming nitric acid (WFNA/RFNA). Moreover, it is characterized by a low toxicity and vapor pressure. Additionally, hydrogen peroxide offers benign decomposition products oxygen and water. [2] As suitable fuel candidates for bipropellants with HTP certain ionic liquids were identified. An overview over this substance class as well as previous work on this topic is given hereafter.

2. Ionic Liquids

2.1 General Definition

Ionic liquids (ILs) are usually defined as salts that melt below 100 °C and consist of an organic or inorganic anion and a sterically demanding, asymmetric organic cation. The beneficial characteristics of ionic liquids such as high thermal stability, low vapor pressure and manifold design possibilities make them promising candidates for new propellants. [3] Recently, thiocyanate based ionic liquids were identified as excellent fuels for hypergolic propellants with hydrogen peroxide as oxidizer in our working group which will be described in detail in the next section.

2.2 Previous Work on Thiocyanate Ionic Liquids at DLR Lampoldshausen

In 2021, the first article concerning ionic liquids with thiocyanate anions reacting hypergolically with hydrogen peroxide was published by Lauck *et al.* [4] Here, the fuel combination HIP_11 was presented, which is a combination of 95 wt% [Emim][SCN] and 5 wt% [Cu][SCN]. This 1st generation fuel has already been successfully demonstrated in a thruster recently. [5] In order to optimize the hypergolic ignition properties of the propellants – with the aim of being able to avoid the copper additive in subsequent fuel generations – investigations on the role of the cation structure on hypergolicity have been carried out. [6] Recent work has shown that protic imidazolium cations induce particularly rapid ignition. One outcome of these investigations is the fuel HIM_35, which is a combination of 65 wt% [Emim][SCN] and 35 wt% [Him][SCN] and exhibits an ignition delay of < 20 ms without the addition of metallic additives. [7,8]

Table 1. Specific impulses and ignition delay times of previous investigated ionic liquid (combinations) as fuels with HTP as oxidizer.

	[Emim][SCN] [4]	**HIP_11** [4] 95 wt% [Emim][SCN] + 5 wt% [Cu][SCN]	**HIM_35** [8] 65 wt% [Emim][SCN] + 35 wt% [Him][SCN]
$I_{sp\,max}$ [s]	317	tbd	314
$\rho\ I_{sp\,max}$ [s·g·cm^{-3}]	430	tbd	429
IDT [ms]	28.3	13.9	16.7

In the above-mentioned previous work, the main objective was the reduction of the ignition delay of the bipropellants. In this work, this aspect will also be investigated on the basis of two selected additives, but the main focus will be on their influence on the specific impulse or, in particular, on the volumetric specific impulse. A high volumetric specific impulse is one of the most critical parameters for a propellant since smaller propellant tanks are required and the spacecraft has a lower overall mass or can carry a higher amount of payload.

3. Combustion Data

3.1 Enthalpy of Formation: Experimental Determination via Bomb Calorimetry

For calculation of the combustion performance of the propellants, enthalpy of formation of the ionic liquids had to be determined. For that purpose, the heat of combustion was measured using an IKA C 200 auto bomb calorimeter according to a standard procedure. Three measurements were carried out for each substance. Because of the presence of nitrogen and sulfur, nitric acid and sulfuric acid were formed by dissolving the combustion products in water (dissolved carbon dioxide was removed by vigorous stirring beforehand). The total acidity was determined by titration with 0.1 M NaOH(aq) for each experiment. Following the procedure of Zaitsau *et al.* [9], the amount of sulfuric acid was derived from the weighed-in mass. By subtraction from the determined value for total acid, the amount of nitric acid was determined. To compensate the formation of nitric acid formed from oxygen, nitrogen and water, the nitrogen correction was performed with a value of -59.7 kJ/mol for 0.1 mol/l aqueous solution of nitric acid. [9,10] Standard molar enthalpies of formation of $H_2O_{(l)}$ (-393.51 kJ/mol) and $CO_{2(g)}$ (-393.51 kJ/mol) at a temperature of 22.0 °C as well as the enthalpy of formation of solution of sulfuric acid $H_2SO_4 \cdot 115\ H_2O_{(aq)}$ (-887.811 kJ/mol) were extracted from literature. [9–11]

3.2 Calculation of Theoretical Performance

Out of the heats of formation which were determined by the previous described method, calculations of the specific impulse (I_{sp}) were carried out using the NASA CEA (Chemical Equilibrium with Applications) code. [12] Basis of these were a 400 N thruster by *Ariane Group* with a chamber pressure of 10 bar and an expansion ratio ε of the nozzle of 330. Moreover, frozen supersonic expansion was assumed. [4,13] The ratio of oxidizer to fuel (ROF) was calculated from the ratio of oxidizer mass flow ($\dot{m}_{fuel}$) and fuel mass flow ($\dot{m}_{oxidizer}$) using the following equation (1):

$$ROF = \frac{\dot{m}_{oxidizer}}{\dot{m}_{fuel}} \tag{1}$$

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For determination of the density specific Impuls ($\rho\ I_{sp}$), the overall density of the propellant at the respective ROF was calculated. For this, density of the oxidizer hydrogen peroxide ($\rho_{oxidizer}$) and the density of the respective ionic liquid fuel (ρ_{fuel}) were included in equation (2) as:

$$\rho_{propellant} = \frac{\rho_{oxidizer} \times \rho_{fuel} \times (ROF+1)}{\rho_{oxidizer} + \rho_{fuel} \times (ROF)} \tag{2}$$

3.3. Drop Test Set-up

In order to determine the ignition delay times (IDTs) of the hypergolic propellants, a drop test set-up as illustrated in Figure 1 was utilized. Here, the IDT is defined as the time span between first contact of oxidizer and fuel and the first visible flame. For determination of this time span, the experiments were recorded by a *Photron fastcam SA-X2* color high-speed camera, with a frame rate of 3000 fps. The several videos were then analyzed frame per frame to determine the desired quantity. For each substance, at least three drop tests were conducted out of which the average value as well as the standard deviation (SD) were calculated. For safety reasons, the experiments were conducted in a fume hood. Further, a quenching mechanism was installed in form of an additional syringe filled with water and portable glass windows were placed in front of the set-up.

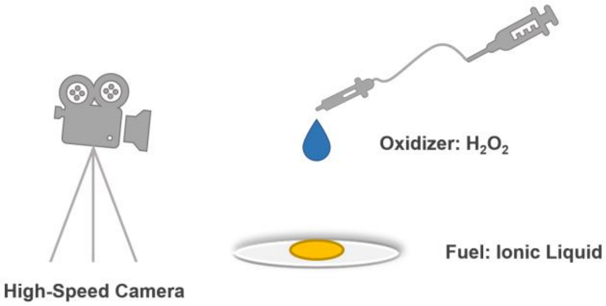

Figure 1. Drop test setup for determination of ignition delay times.

4. Results and Discussion

4.1 Fuel Additives : Selection and Synthesis

In a publication by Sun and Tang, nitrogen-rich ionic liquids with dicyanamide anions were investigated as additives for an ionic liquid with this anion. [14] In addition to an increase in density due to the additives, an increase in specific impulse was obtained. However, this type of fuel reacts hypergolically with the oxidizer WFNA which is somewhat less advantageous compared to highly concentrated hydrogen peroxide, as discussed in section 1.1. For this reason, additives composed of nitrogen-rich organic cations and thiocyanate anions are investigated in this work.

As base substance, the RTIL (room temperature ionic liquid) [Emim][SCN] was chosen. This IL was already characterized as suitable fuel basis for hypergolic ionic liquids with HTP, which is available for purchase. [4] Two thiocyanate-based additives [Gua][SCN] (guanidnium thiocyanate) and [Ch][SCN] (choline thiocyanate) were selected for the following investigations. The selection was based on the atomic composition of the cations and the physical and chemical properties of the two substances. While the substance [Gua][SCN] is commercially available and was purchased by *Carl Roth*, [Ch][SCN] was synthesized in the physical-chemical laboratory.

4.1.1 Synthesis of [Ch][SCN]

12.9 g (92.4 mmol) choline chloride was dissolved in 100 ml acetone and 100 ml isopropyl alcohol. 9.88 g (102 mmol) of potassium thiocyanate was added and the mixture was stirred at room temperature for 16 h. The formed potassium chloride was filtered off and the product was evaporated to dryness. 9.70 (59.8 mmol; ≙ 65 %) of [Ch][SCN] was obtained and characterized via NMR and IR spectroscopy. Purity of the substance was validated through ion chromatography (anionic and cationic method) and Karl Fischer titration.

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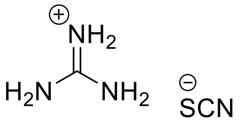
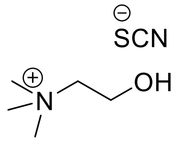
Figure 2 shows a photography of the purchased liquid IL [Emim][SCN] (left) together with the solid additives [Gua][SCN] (middle) and the synthesized compound [Ch][SCN] (right).

Figure 2. Photography of the pure substances: left: [Emim][SCN], middle: [Gua][SCN], right: [Ch][SCN].

4.2 Characterization of pure additives

Table 2 contains an overview of physical and chemical properties of [Gua][SCN] and [Ch][SCN] as well as calculated performance. Enthalpies of formation were determined by the method described in detail in section 3.1. On that basis, the maximum theoretical specific impulse at the respective ROF were calculated with NASA CEA, as outlined in chapter 3.2. Density of [Ch][SCN] was measured with an Ultrapyc 5000 Micro gaspycnometer by Anton Paar at 25 °C, the density of [Gua][SCN] was taken from its safety data sheet. [15]

Table 2. Physical and chemical properties of [Gua][SCN] and [Ch][SCN] as well as calculated combustion data.

	[Gua][SCN]	[Ch][SCN]
chemical structure	 [Gua][SCN]	 [Ch][SCN]
ΔH_f [$\mathrm{kJ\cdot mol^{-1}}$]	-115	184
$I_{sp\ max}$ [s]	299	327
ROF @ $I_{sp\ max}$	2.5	3.4
T_c @ $I_{sp\ max}$ [K]	2663	2889
ρ [$\mathrm{g\cdot cm^{-3}}$] 25 °C	1.29 (20 °C) [15]	1.164 (25 °C)
$\rho\ I_{sp\ \mathrm{max}}$[$\mathrm{s\cdot g\cdot cm^{-3}}$]	417	446

4.2.1 Theoretical Performance of the Additives

On the left side of Figure 3, the calculated specific impulses of [Ch][SCN] (blue) and [Gua][SCN] (green) together with 98 wt% H_2O_2 are shown over ROFs from one to six. The difference between the two curve progressions is significant, especially with a view of the respective maxima. The $I_{sp\ \mathrm{max}}$ of [Gua][SCN] of 299 s is lower compared to the base IL [Emim][SCN] with a calculated $I_{sp\ \mathrm{max}}$ of 317 s. Therefore, an improvement of the theoretical performance can unlikely be achieved through addition of this compound. In contrast to that, [Ch][SCN] offers a theoretical maximum of I_{sp} of 327 s under these conditions. Therefore, with this substance an enhancement of the I_{sp} of the fuel is expected, which will be discussed in the next chapter. The variation of the calculated data of the two substances originates from their different atomic composition, which especially result in their unequal enthalpies of formation.

The diagram on the right side of Figure 3 shows the comparison of the volumetric specific impulses over the respective ROFs for the two pure substances with HTP. In terms of the volumetric specific impulses, [Ch][SCN] shows a significantly higher value of 447 $\mathrm{s\cdot g\cdot cm^{-3}}$ compared with the one of [Gua][SCN] of 417 $\mathrm{s\cdot g\cdot cm^{-3}}$.

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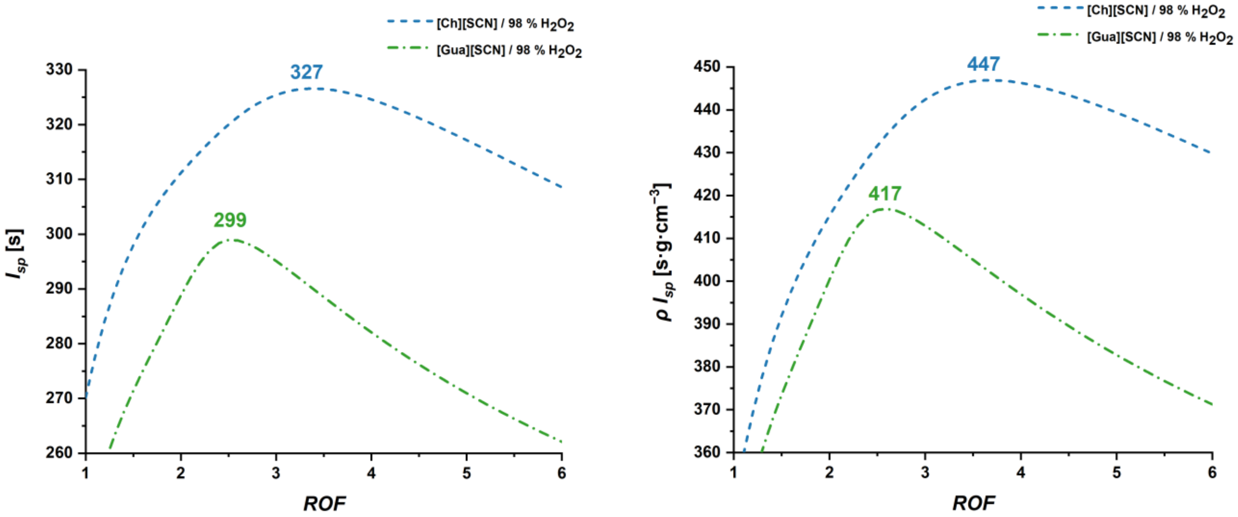

Figure 3. Comparison of I_{sp} (left) and ρ I_{sp} (right) over *ROF* of [Ch][SCN] (blue) and [Gua][SCN] (green) with 98 wt% H_2O_2 as oxidizer.

4.2.2 Evaluation of Hypergolic Drop Tests

The ignition delay times of both substances were determined according to the method described in section 3.3. In Table 3, the ignition delay times (IDTs) of the thiocyanate compounds as fuels with HTP (~97 wt%) as oxidizer are listed. The determined IDT of [Gua][SCN] of 37.7 ms is clearly shorter than the one of [Ch][SCN] of 62.4 ms. Even though both additives have a longer IDT than the base fuel [Emim][SCN], [Gua][SCN] is expected to have a less negative effect when dissolved in EMIM SCN. Therefore, the ignition behavior of only [Gua][SCN] as additive is investigated in 4.3.3., experiments with [Ch][SCN] will follow. In Figure 4, sequences of a high-speed video of a drop test with [Ch][SCN] as fuel are shown.

Table 3. Ignition delay times of [Gua][SCN] and [Ch][SCN] with HTP (~97 wt%) and standard deviations of the latter (SD of [Gua][SCN] was not determinable due to an insufficient number of ignitions).

	[Gua][SCN]	[Ch][SCN]
IDT [ms]	37.7	62.4
SD IDT [ms]	-	5.3

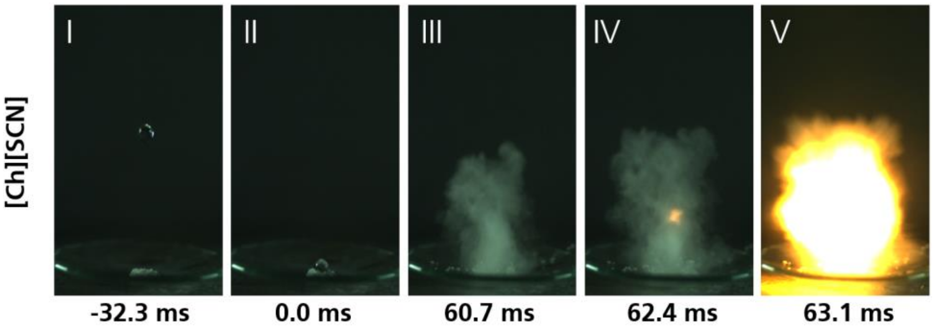

Figure 4. Single sequences of a high-speed video of a hypergolic drop test. Oxidizer: HTP, Fuel: [Ch][SCN].

4.3 Hypergolic Ignition Behavior and Performance of Mixtures

4.3.1 Preparation and Characterization of the mixtures

For the reasons given above, [Emim][SCN] was chosen as base fluid for the fuel mixtures. In order to characterize these mixtures, the solubility limit of the additives at room temperature were evaluated. Figure 5 shows a photography of the solubility test of [Gua][SCN] in [Emim][SCN]. As this picture shows, the maximum soluble amount of [Gua][SCN] lies within 30 wt% and 40 wt%. For further experiments and calculations, an amount of 30 wt% was set for both additives.

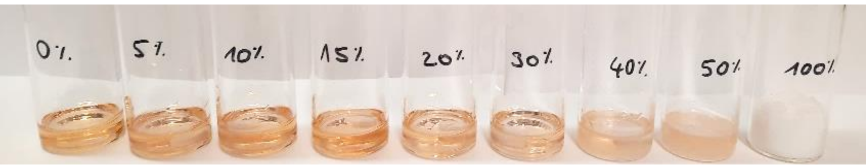

Figure 5. Photography of different solutions of [Gua][SCN] in [Emim][SCN]. 0% (left): pure [Emim][SCN]; 100% (right): pure [Gua][SCN].

Figure 6 shows the results of the viscosity and density measurements of the mixtures of [Gua][SCN] in [Emim][SCN]. Density measurements were conducted with a density meter Easy D40 by Mettler-Toledo at 25 °C, viscosity measurements were conducted using LOVIS 2000 M micro viscosimeter by Anton Paar, also at 25 °C. As expected, both quantities increase linearly with increasing proportion of [Gua][SCN]. In the viscosity curve (green triangles), a bend can be seen at low percentages which may have been caused by inaccuracies in weighing or measurement. A smoother linear curve is obtained from the density measurements (blue squares). The high density of the blends with high additive content may be beneficial to the volumetric specific impulse, as will be discussed in the next chapter.

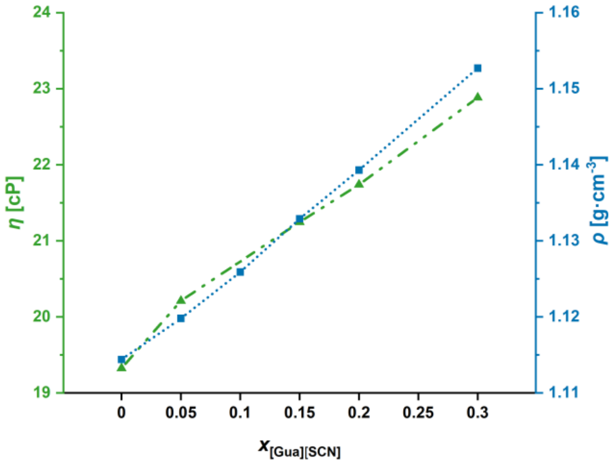

Figure 6. Dependency of viscosity (green) and density (blue) of various mixing ratios of [Gua][SCN] in [Emim][SCN].

Table 4. Viscosity and density data of [Emim][SCN], 70 % [Emim][SCN] + 30 % [Gua][SCN] and 70 % [Emim][SCN] + 30 % [Ch][SCN] compared with MMH.

	[Emim][SCN]	70 % [Emim][SCN] + 30 % [Gua][SCN]	70 % [Emim][SCN] + 30 % [Ch][SCN]	MMH
η_{fuel} [cP]	20.5	22.9	33.8	0.85[16]
ρ_{fuel} [g·cm^{-3}]	1.114	1.153	1.112	0.88[16]

4.3.2 Theoretical Performance of Mixtures

In Table 5, calculated performance of the hypergolic propellant combinations is summarized together with the theoretical data of the conventional propellant combination MMH and NTO. The comparison with the initial substance

[Emim][SCN] shows, as expected, that the addition of 30 wt% [Gua][SCN] results in a slight reduction of the calculated values for both $I_{sp\ \mathrm{max}}$ and $\rho\ I_{sp\ \mathrm{max}}$. However, the addition of [Ch][SCN] achieved the desired effect: Both the calculated $I_{sp\ \mathrm{max}}$ and $\rho\ I_{sp\ \mathrm{max}}$ were increased. The fact that the percentage of reduction and increase of the values is not very high is attributable to the fact that the high ROF gives the influence of the fuel component much less weight than that of the oxidizer component. Nevertheless, it is worth noting that it was possible to show that additives based on organic salts can increase the specific impulse by several seconds.

Table 5. Calculated combustion data and density of [Emim][SCN], 70 % [Emim][SCN] + 30 % [Gua][SCN] and 70 % [Emim][SCN] + 30 % [Ch][SCN] with 98 wt% H_2O_2 as oxidizer compared with MMH / NTO.

	[Emim][SCN] / HTP	70 % [Emim][SCN] + 30 % [Gua][SCN] / HTP	70 % [Emim][SCN] + 30 % [Ch][SCN] / HTP	MMH / NTO
$I_{sp\ max}$ [s]	317	313	320	337
ROF	3.8	3.4	3.7	1.7
$\rho\ I_{sp\ max}$ [s·g·cm^{-3}]	430	427	434	398

A visualization of the full I_{sp} data over ROF is shown on the left of Figure 7, next to the respective $\rho\ I_{sp}$ curves on the right. The maximum I_{sp} of the fuel mixture with [Ch][SCN] as additive (blue curve) of 320 s is higher than the one with [Gua][SCN] of 313 s. As MMH / NTO has a maximum I_{sp} of 337 s, both IL-based fuels don't meet this benchmark. When it comes to the volumetric specific impulse, the picture looks different. As shown on the right of Figure 7, both mixtures exceed the calculated maximum value of MMH /NTO and are therefore more advantageous in this regard.

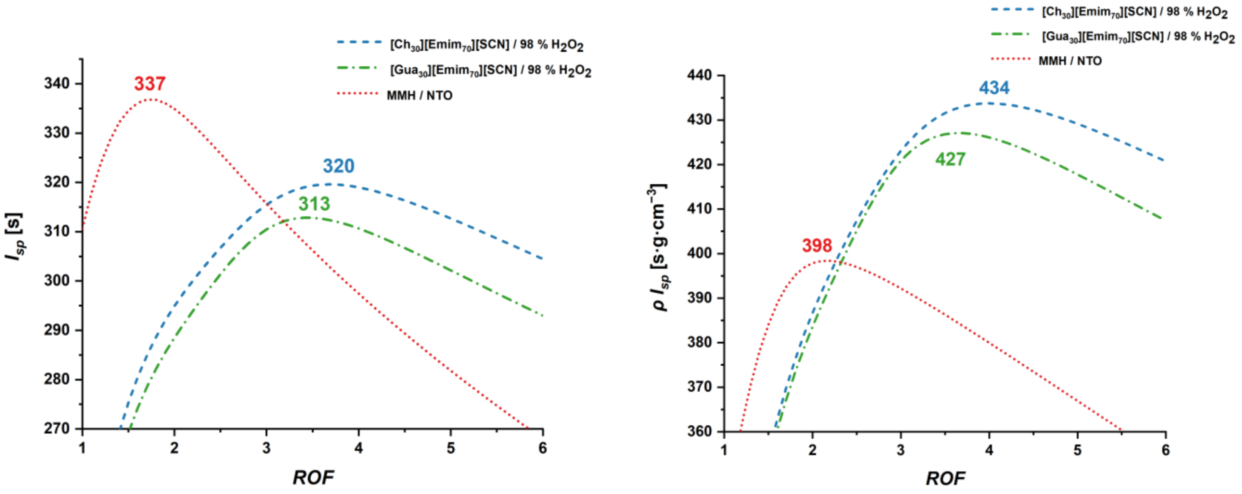

Figure 7. Comparison of I_{sp} (left) and $\rho\ I_{sp}$ (right) over ROF of [Ch$_{30}$][Emim$_{70}$][SCN] (blue) and [Gua$_{30}$][Emim$_{70}$][SCN] (green) with 98 wt% H_2O_2 as oxidizer and MMH / NTO (red).

4.3.3 Evaluation of Hypergolic Ignition in Drop Tests

In Table 6, the average ignition delay times of [Emim][SCN] and the mixture of 70 % [Emim][SCN] and 30 % [Gua][SCN] are compared.

Table 6. Ignition delay times of [Emim][SCN] and 70 % [Emim][SCN] + 30 % [Gua][SCN] with HTP (~97 wt%) along with the regarding standard deviations.

	[Emim][SCN] / HTP	70 % [Emim][SCN] + 30 % [Gua][SCN] / HTP
IDT [ms]	28.3	26.5
SD IDT [ms]	0.3	3.3

Very interestingly, the average IDT of the [Gua][SCN] containing fuel was lower than that of pure [Emim][SCN] – despite a clearly higher IDT of the pure solid. Noticeably, the viscosity of this mixture is not enhanced significantly compared to the pure IL (Table 3), but the viscosity of the mixture is still higher – which usually adversely affects the

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IDT. In order to gain more information about this issue, IDTs of the above characterized mixtures with various amounts of [Gua][SCN] were investigated by hypergolic drop tests. Figure 8 shows the average IDTs of these mixtures with as well as the ones of the two pure substances. The IDTs of mixtures containing 5 wt% and 10 wt% [Gua][SCN] are increased, as expected. However, with further growing percentage of [Gua][SCN] a reciprocal reduction of the IDT is observed. The cause of this behavior is not yet fully clarified but will be subject of further investigations.

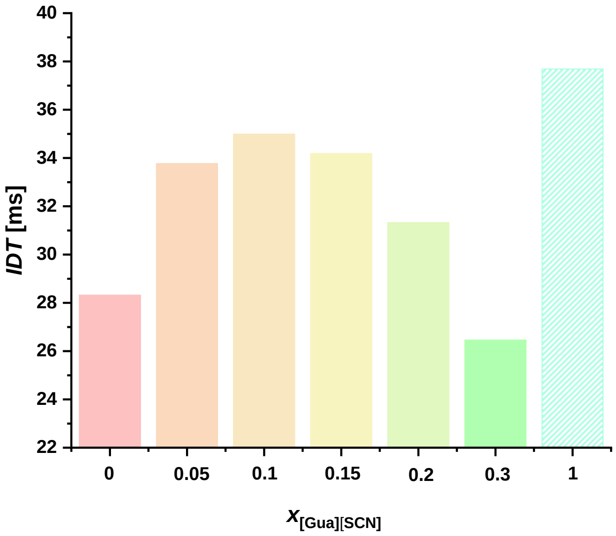

Figure 8. IDTs of fuels with various mixing ratios of [Gua][SCN] in [Emim][SCN]; oxidizer: H_2O_2 (~97 wt%).

5. Conclusion and Outlook

In this work, two different thiocyanate-based salts were used as additives for fuels which, together with hydrogen peroxide as an oxidizer, form "green" hypergolic propellants. In the selection of the fuels, the main focus was on the functional groups containing nitrogen as well as oxygen. When the pure additives were investigated, it was found that [Gua][SCN] exhibited a faster hypergolic ignition behavior, whereas a significantly higher specific impulse was calculated for [Ch][SCN].

These properties were also evident in the same way in the blends of the additives with the liquid IL [Emim][SCN]: While weaker performance data was calculated in the fuel containing 30 wt% [Gua][SCN], interestingly, even a decrease of the ignition delay was obtained here compared to the base fluid. Further work will address the subject, whether additional amino groups as substituents on the guanidinium cation may have a positive effect on the standard enthalpy of formation and thus on the calculated combustion data.

In contrast, with the fuel mixture containing 30 wt% [Ch][SCN], the initial aim could be accomplished, as both an increase in I_{sp} and $\rho\ I_{sp}$ was calculated with HTP in comparison to the base fluid. Since [Ch][SCN], unlike [Gua][SCN] and other additives investigated so far, contains an OH-moiety, it will be further investigated whether this functionality causes the favorable behavior of this additive.

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