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Hypergolic Propellants with Hydrogen Peroxide: Carbon-Based Nanomaterials as Additives in Ionic Liquid Fuels

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Abstract
As a part of the DLR internal project *NatAs 2022* (nanoparticles in propulsion systems), carbon-based nanomaterials were investigated as additives to explore their positive influence on the properties of ionic liquid-based propellants and their ability to form so-called heat transfer fluids (HTFs). The physical and chemical properties of the new HTFs such as density, viscosity, temperature stability and oxidation behavior were investigated. This was followed by analysis of hypergolic ignition and determination of the surface tension. The most important result is that an increase in thermal conductivity by 28 % and in heat capacity by 26 % was achieved, which is an important step in the development of this type of heat transfer fluids.

1. Introduction

1.1 Green Propellants

Hydrazine-based propellants are still state of the art for space vehicles like satellites, space probes or upper stages. However, hydrazine as well as its commonly used derivatives such as MMH (monomethyl hydrazine) or UDMH (unsymmetrical dimethylhydrazine) are classified as toxic and carcinogenic. [1] Not only does this make them hazardous to handle, but also leads to high costs in fueling spacecrafts with these propellants due to the need of special protective equipment. As fuels for hypergolic propellants hydrazine is often combined with dinitrogen tetroxide as oxidizer. This substance is also extremely toxic, to the point of being lethal if inhaled. [1]

For these reasons, research is conducted worldwide to find alternatives that could replace these propellants in the future. The present work focuses on hypergolic combinations with hydrogen peroxide as oxidizer. Hydrogen peroxide has proven to be advantageous due to its low toxicity, low vapor pressure and high performance. [2] As fuel component ionic liquids with thiocyanate anions are being developed. Ionic liquids (ILs) are salts with organic cations or anions with melting points below or around 100 °C. [3] Their favorable properties, such as negligible vapor pressure and high density, make them particularly interesting as fuel candidates. In recent years, the development of these propellant combinations has progressed at the DLR Institute of Space Propulsion in Lampoldshausen. [4–6]

1.2 Nanomaterials

The prefix "nano" is derived from the ancient Greek word "nanos" meaning "dwarf" and refers to the 10^{-9th} part of a physical unit. By definition, nanoparticles are a group of particles with a size between 1 nm and 100 nm. [7] Many nanomaterials differ from the corresponding bulk material in their electrical, mechanical and optical properties. This can be attributed to the extremely high surface-to-volume ratio of nanostructures which also leads to a drastically increased reactivity. Therefore, these materials find many applications in the field of catalysis. Meanwhile, there is a large number of characterized nanomaterials of different types, shapes and sizes. There are also many different ways of synthesizing nanomaterials, which can be basically divided into top-down and bottom-up methods. [8,9] In Figure 1, the number of publications under the keyword "nanoparticles" on the SciFinder database is plotted since the year 1990. This figure illustrates the huge increase in interest in this area of research in recent decades.

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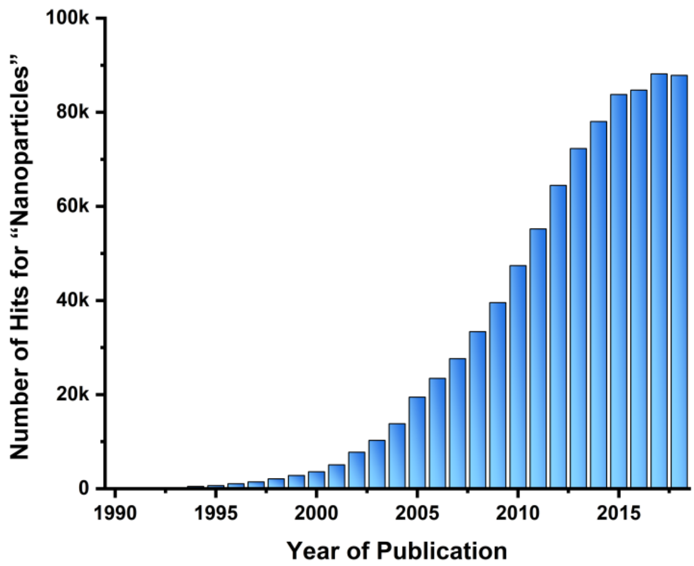

Figure 1. Publications for the keyword "nanoparticles" fount at the Scifinder database since 1990.

1.3 Heat Transfer Fluids (HTFs)

A heat transfer fluid (HTF) is a liquid which is able to transport thermal energy. This property allows HTFs to be used for cooling and heating in a variety of applications. Important properties for HTFs are generally: [10]

- Good thermal conductivity
- High thermal stability
- Low viscosity
- Low solidification or melting point
- Non-corrosive to piping
- Low toxicity
- Environmentally friendly
- Low cost
- Safe and easy to handle

In principle, pure ILs without the addition of nanoparticles can also be used as HTFs, but the various fluids have different suitability for this application. The properties of HTFs as thermal stability, heat capacity, thermal conductivity of corrosivity can be modified or optimized by the addition of suitable nanoparticles. [11–14]

2. Selection of Carbon-Based Nanomaterials

Many different types of nanomaterials can be used to modify the properties of ionic liquids (ILs), which will serve as base fluid in this project. One criterion for classification is the distinction between metallic and non-metallic nanomaterials. In the following, only non-metallic nanoparticles will be considered initially, as these fuel additives provide the decisive advantage that only gaseous combustion products should be formed when the fuel is ignited. In case of metallic components, the combustion products include metal oxides. For satellite and orbital propulsion systems, this means that these solid combustion products can deposit on the solar panels of the spacecraft and impair their performance. Another disadvantage of compounds containing heavy transition metals is that they tend to have a negative effect on the specific impulse of the propellants due to their high molecular weight combustion products.

Carbon-based nanoparticles can influence ionic liquids in a variety of ways. For example, it is already known that they are able to increase the thermal conductivity of ILs, to influence melting points as well as to have a positive effect on hypergolicity. [15,16] In addition, some nanoparticles have already been shown to have a viscosity-lowering effect on ionic liquids, especially for dispersions with low concentrations. [16–18] This is of great interest for hypergolic propellants, as a reduced viscosity could not only facilitate pumping and spraying of the propellant, but is also likely

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to shorten the ignition delay due to faster mixing with the oxidizer. In addition, carbon-based particles are not expected to have a negative effect on specific impulse due to their light combustion products.

An important consideration for selection of the nanoparticles was their ability to reduce the viscosity of the ionic liquids upon their addition. In terms of the viscosity reducing properties of ionic liquids, several promising nanomaterials have been found in literature, which will be discussed in more detail below:

- Untreated multi-walled carbon nanotubes (MWCNTs)
- Carboxylated multi-walled carbon nanotubes (oMWCNTs)
- Sulfuric acid treated multi-walled carbon nanotubes (sMWCNTs)
- Graphite-like carbon nitride nanoplatelets (GCNNs)
- Carboxylated detonation nanodiamonds (DNDs)
- Carboxylated graphene nanoplatelets (oGr-NPs)

A viscosity-lowering effect has already been observed for carboxyl-functionalized multi-walled carbon nanotubes (oMWCNTs) in ionic liquids. [17] Dispersions with low weight fractions (< 1 wt%) of carboxyl-functionalized CNTs (carbon nanotubes) led to a decrease in viscosity of imidazole-based ILs by more than 70 percent. The decrease in viscosity was attributed to two reasons by Neo and Ouyang:[17] The first one is the formation of hydrogen bonds between the cations and the acid groups on the CNTs, leading to the formation of larger effective cations and a consequent increase in size disparity between cation and anion. The second possible reason, according to the authors, is the increased disorder in the ionic structure caused by the nanomaterials. Carbon nanotubes with different surface functionalizations were investigated, as these are crucial for the interactions with ILs and thus presumably also for the influence on viscosity.
Graphene nanoplatelets (Gr-NPs) were also shown to decrease the viscosity of ILs at low mass percentages. [16,19,20] This is explained by a perturbation of the order of the ion network.
Similarly, in dispersions containing carboxylated detonation nanodiamonds (DNDs), a sometimes significant viscosity reduction (over 90%) has been achieved at very low weight percentages.. [18,21]
Graphite-like carbon nitride nanoplatelets (GCNNs) are also promising candidates, since they have a similar structure to graphene nanoplatelets and may therefore have a similar effect on viscosity. In addition, their catalytic effect in the decomposition of hydrogen peroxide is known, suggesting a possible positive effect on ignition delay. [22]

Within a pre-screening, all of the above-mentioned carbon nanoparticles were tested in two thiocyanate-based ionic liquids. None of the selected nanomaterials was able to reduce the viscosity of the tested thiocyanate-based ionic liquids. Finally, oGr-NPs (carboxylated graphene nanoplatelets) were selected for further investigation as they did not increase the viscosity of the chosen ionic liquids in the tested concentrations and were commercially available. The oGr-NPs were purchased from Sigma Aldrich and used as received. Figure 2c shows the platelet shaped structure of the particles. The stoichiometric composition (89.6% carbon, 10.4 % oxygen) was measured via EDX (energy-dispersive X-ray) spectroscopy. The size of the particles with hydrate shell was determined by DLS to be 127.7 nm ± 13.8 nm.

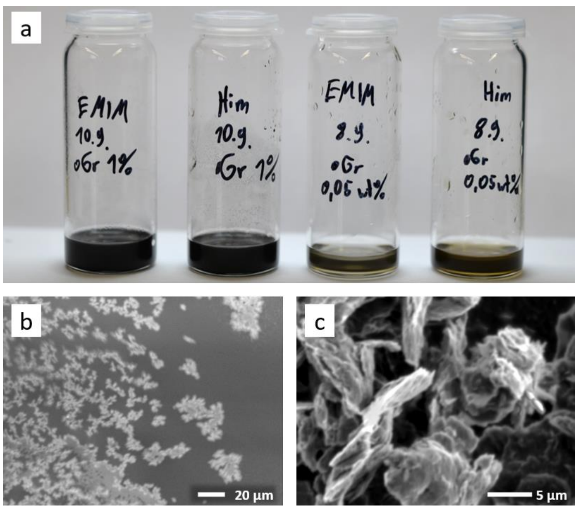

Figure 2: 1 wt% and 0.05 wt% suspensions of oGr-NP in [Emim][SCN] und [Him/Emim][SCN]; b: SEM-picture of oGr-NP in [Emim][SCN]; c: SEM-picture oGr-NP on carbon tape.

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3. Nanofluids: Preparation, Characterization and Testing

3.1 Selection of the base fluid

The IL-based fuel HIM_35 was selected as base fluid. It is composed of the liquid IL [Emim][SCN] (65 wt%) and the solid IL component [Him][SCN] (35 wt%). The liquid fuel reacts hypergolically with highly concentrated hydrogen peroxide. [6] HIM_35 is thus a promising fuel for future applications, with the advantage that neither transition metal nor boron or hydride compounds are used.

First successful hot fire tests with HIM_35 were conducted at DLR Lampoldshausen recently. In more advanced variants of the engine, either the oxidizer or the fuel component could be used as a cooling medium. In case of the fuel component, it will be investigated whether the properties of HIM_35 as a so-called "heat transfer fluid" can be optimized with the aid of nanomaterials.

For preparation of the base fluid HIM_35, 21 ml [Emim][SCN] (purchased from IoLiTec GmbH) was first dried for 3 h on a rotary evaporator to remove water residues. Subsequently, 12.6 g of [Him][SCN] (Synthesis of *CTT; department of Chemical Propellant Technology*) was dissolved by stirring at RT.

After addition of oGr-NP (polycarboxyl functionalized graphene nanoplatelets, Sigma-Aldrich), the solutions were treated with the ultrasonic probe, using the following program: Power: 40%, Time: 2 min, Intensity: 0.5 Cycles. This step is used to disperse the nanoparticles, but for comparability the procedure was also performed with the pure HIM_35 solution. A photo of the three samples immediately after the preparation process is shown in Figure 3.

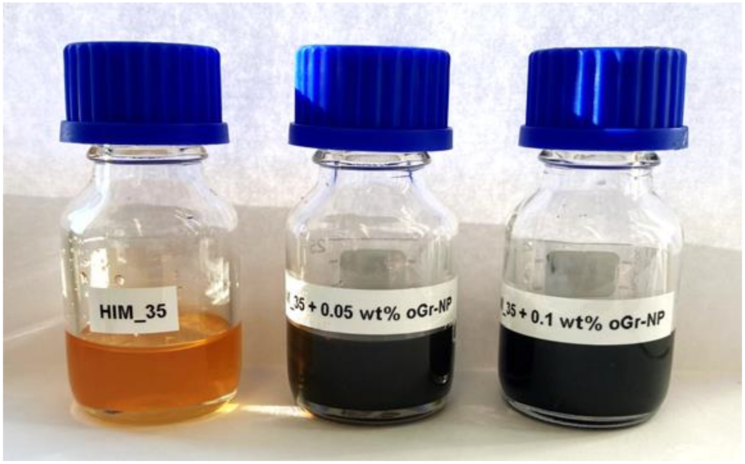

Figure 3: Base fluid HIM_35 (left) with 0.05 wt% (middle) and 0.1 wt% (right) oGr-NP as additives.

3.2 Physical and Chemical Properties

In the first step, the density and viscosity of the three test fluids were determined. The average values are summarized in Table 1.

Table 1: Overview of the density and viscosity of the nanofluids, each at a temperature of 25 °C.

	HIM_35	**HIM_35 + 0.05 wt% oGr-NP**	**HIM_35 + 0.1 wt% oGr-NP**
Density [g·cm^{-3}]	1.1541	1.1544	1.1547
Viscosity [mPa·s]	49.9	50.6	51.2

Figure 4 contains a graphical representation of these measured values. It is clear that the nanoparticles have no significant influence either on the density or the viscosity of the substances.

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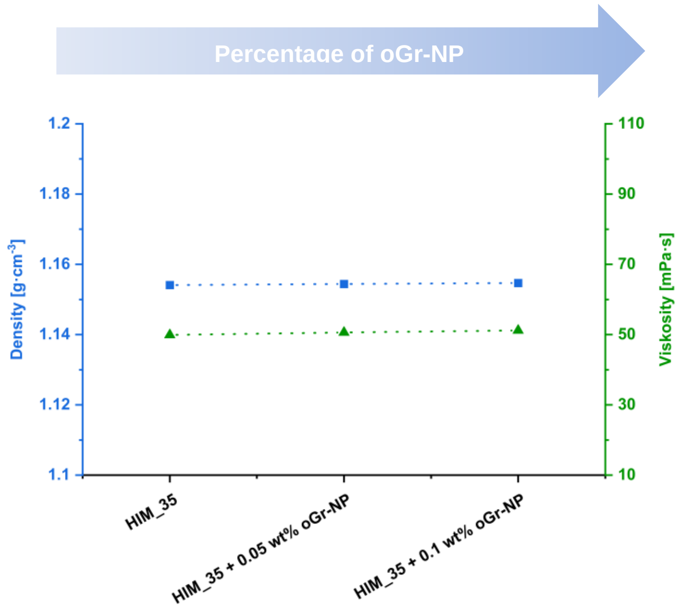

Figure 4. Diagram of the density and viscosity of the nanofluids, each at a temperature of 25 °C.

3.4 FTIR-Spectroscopy

To characterize the test fluids, FTIR spectra shown in Figure 5 were recorded. Apart from the asymmetric stretching vibration of the CO_2 contained in the air at ν_{as} = 2349 cm^{-1}, which is pronounced to different degrees, the characteristic vibrational bands of the three substances are very similar. Especially the orange marked *ν(S-C≡N)* = 2045 cm^{-1} bands of the thiocyanate anions [23] and the yellow highlighted *ν(N-H)* and *ν(C-H)* vibrations of the imidazolium-based cations are to be emphasized as characteristic bands.

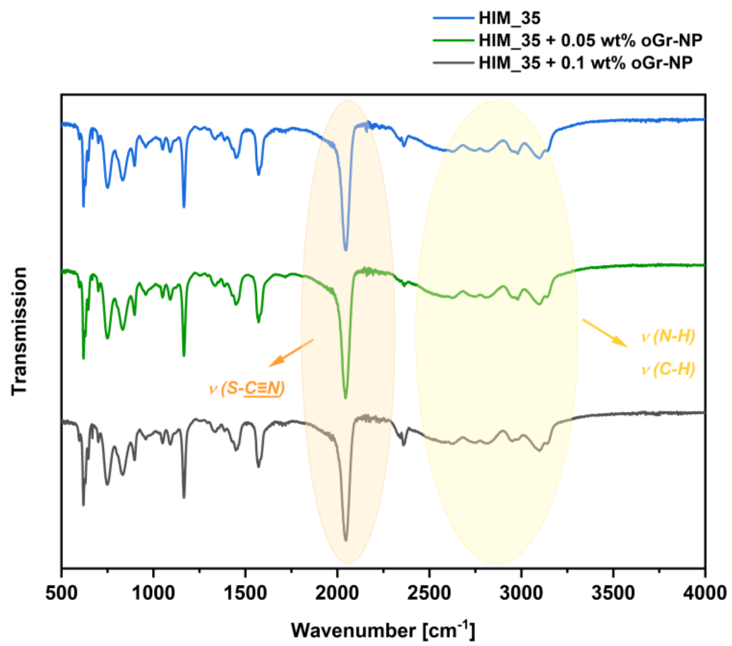

Figure 5. Comparison of FTIR-spectra of HIM_35 and the two nanofluids.

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3.4 TG/DSC-Analysis

3.4.1 Measurements in the Temperature Range from 30 °C to 600 °C under N_2 Atmosphere

TG and DSC analyses were carried out in the temperature range 30 °C – 600 °C in Al crucibles under N_2 atmosphere. The heating rate was 10 K/min. Table 2 summarizes the decomposition temperatures of the three investigated fluids. The values were determined from the 5%-onset of the TG curves, whereby the temperatures given are average values formed from two measurements.

Table 2: Decomposition temperatures of the nanofluids determined by TG/DSC analyses in a temperature range from 30 °C to 600 °C under N_2 atmosphere.

	HIM_35	**HIM_35 + 0.05 wt% oGr-NP**	**HIM_35 + 0.1 wt% oGr-NP**
Decomposition Temperature [°C]	256	258	268

Figure 6 shows the TG and DSC diagrams of the measurements described above. The course of the two curves is very similar for the three substances in each case. A slight increase in the determined decomposition temperature was obtained with increasing proportion of oGr-NP: from 256 °C for pure HIM_35 via 258 °C for 0.05 wt% oGr-NP to 268 °C for 0.1 wt% oGr-NP.

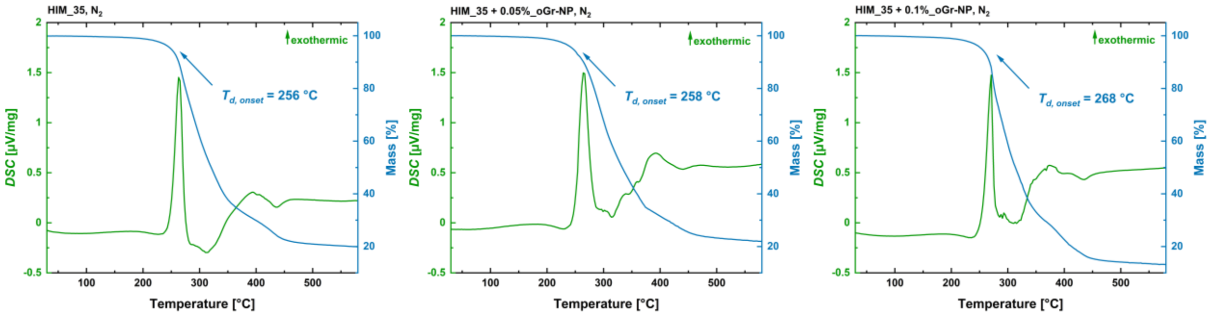

Figure 6. TG and DSC diagrams of HIM_35 and nanofluids with 0.05 and 0.1 wt% oGr-NP in Al crucibles under N_2 gas in a temperature range between 30 °C and 600 °C.

3.4.2 Measurements in the Temperature Range from 30 °C to 600 °C under O_2 Atmosphere

To investigate the oxidation behavior of the substances, measurements were also carried out in the high-temperature range of 30 °C – 600 °C at a heating rate of 10 K/min under an oxygen atmosphere. The average values determined for the decomposition temperatures are shown in Table 3.

Table 3: Decomposition temperatures of the nanofluids determined by TG/DSC analyses in a temperature range from 30 °C to 600 °C under O_2 atmosphere.

	HIM_35	**HIM_35 + 0.05 wt% oGr-NP**	**HIM_35 + 0.1 wt% oGr-NP**
Decomposition Temperature [°C]	277	282	281

Figure 7 shows the graphs for the measurements under oxygen for each of the three samples investigated. Again, there is a clear analogy between the diagrams of the three substances. All three decomposition temperatures are in a very similar range between 277 °C and 282 °C. No increase in mass was detected in any of the TG curves, indicating that no significant oxidation of the ionic liquids occurred prior to the decomposition reaction.

By comparison of the high-temperature measurements under N_2 and O_2 atmospheres, it is worth noting that all decomposition reactions under N_2 atmosphere are accompanied by an exothermic reaction, whereas an endothermic

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reaction is detected in the DSC signals under O_2 atmosphere. In addition, the decomposition reaction under an oxygen atmosphere takes place at significantly higher temperatures in each case with a temperature difference of around 20 °C compared to the nitrogen atmosphere.

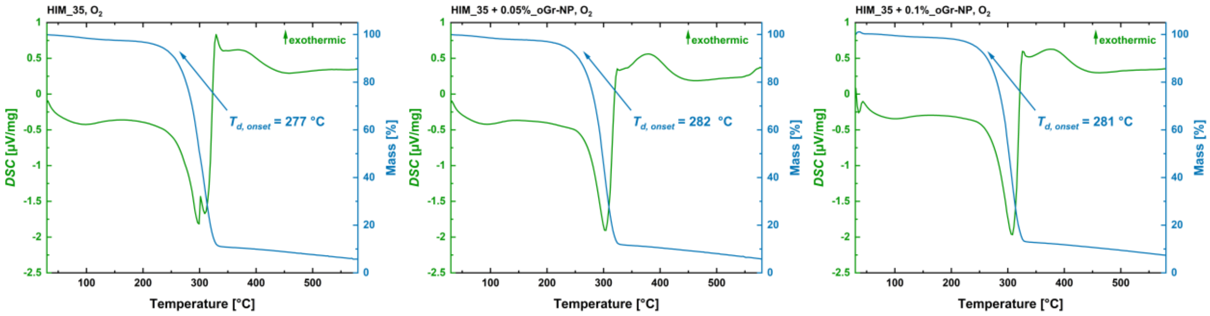

Figure 7. TG und DSC measurement of HIM_35 and nanofluids with 0.05 and 0.1 wt% oGr-NP in Al cruicibles under O_2 gas in a temperature range between 30 °C and 600 °C.

3.4.3 Measurements in the Temperature Range from -100 °C to 50 °C under He Atmosphere

TG and DSC measurements of HIM_35 and the nanofluids with 0.05 and 0.1 wt% oGr-NPs were carried out at low temperatures under He atmosphere in Al crucibles. In the low temperature range, helium was used as inert gas instead of nitrogen because more uniform signals can be obtained here due to the higher thermal conductivity. For this purpose, the Pt-furnace, which is used by default for high temperature measurements, was exchanged by an Ag-furnace and a liquid nitrogen cooling system was installed. Two measurements were performed for each of the three substances. The aim of these investigations was to determine the melting points of the fluids and the influence of the nanoparticles on them. For this purpose, the temperature profile was modified by first cooling the sample from 30 °C to -100 °C at a rate of 5 K/min. This temperature was maintained for 5 min, followed by a temperature increase to 50°C at a heating rate of 5 K/min.

However, no clear signals for crystallization or melting processes could be observed in any of the plots. This can be explained by the fact that upon cooling, the solid [Him][SCN] dissolved in [Emim][SCN] slowly precipitates, followed by a smooth transition to crystallization of the entire solution. The literature value for the melting point of [Emim][SCN] was found to be -6 °C [24]. Figure 8 shows an example of a low-temperature graph of HIM_35. The gray curve indicates the temperature profile. It should be noted here that the DSC curve (green) can only be evaluated in areas of constant temperature slope (highlighted in yellow). The signals observed in the DSC curve are always accompanied by a change in the temperature curve and are therefore not related to the behavior of the sample. There is no significant change in the TG curve (blue) throughout the measurement, indicating that no decomposition or

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evaporation processes are taking place. The TG and DSC diagrams of the samples containing oGr-NPs show similar curves.

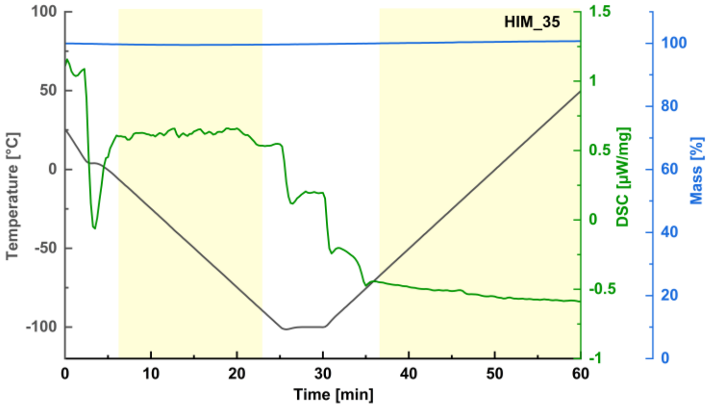

Figure 8: Low temperature TG and DSC measurement of HIM_35 in Al crucibles under He atmosphere.

3.5 Analysis of the Properties of Nanofluids as Heat Transfer Fluids

The next characterization step was measurement of surface tension, thermal conductivity and heat capacity. The average values obtained are summarized in Table 4.

Table 4: Overview of the determined average values of surface tension, thermal conductivity and heat capacity of HIM_35 and the nanofluids at a temperature of approx. 25 °C.

	HIM_35	**HIM_35 + 0.05 wt% oGr-NP**	**HIM_35 + 0.1 wt% oGr-NP**
Surface tension [mN/m]	49.2	49.0	50.3
Thermal conductivity [mW/m·K]	0.0637	0.0817	0.0812
Heat capacity [MJ/ m^{3}K]	1.00	1.22	1.26

Figure 9 contains a graphical representation of the measured values from Table 4. The surface tension of the dispersion of HIM_35 with 0.05 wt% oGr-NP is almost constant compared to the base fluid, the value for 0.1 wt% oGr-NP is slightly higher. Overall, the influence of these nanoparticle fractions on the surface tension can be described as negligible. However, the thermal conductivity and heat capacity increase significantly with addition of 0.05 wt% oGr-NP compared to HIM_35 without additives. While no further increase in thermal conductivity could be observed at 0.1 wt% oGr-NP, the heat capacity increased even further for this nanofluid. Overall, the absolute values for both quantities are rather low compared to other HTFs. Nevertheless, by increasing the values after addition of the oGr-NP, the properties of the ionic liquids were clearly favored for their application as cooling media.

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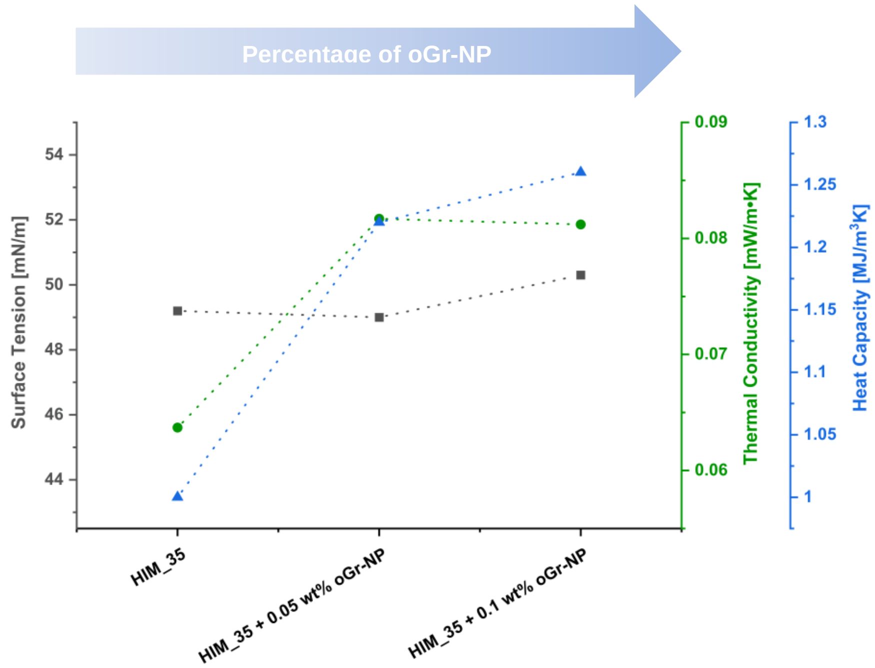

Figure 9: Average values for surface tension (gray), thermal conductivity (green) and heat capacity (blue) of the nanofluids in comparison.

3.6 Impact of Carbon Nanoparticles on the Hypergolic Ignition Behavior

In the previous section, the suitability of nanofluids as HTFs was investigated. An IL-based fluid which serves as a cooling medium in a rocket combustion chamber is expected to also function as propellant after passing through the cooling channels. The base fluid HIM_35 has already been identified as promising hypergolic fuel candidate in combination with highly concentrated hydrogen peroxide as oxidizer. [6] To determine the influence of carbon nanoparticles on the hypergolic ignition behavior, drop tests were performed and filmed by a high-speed camera with a recording speed of 3000 frames per second. Figure 10 shows individual sequences of such a video. The IL-based fuel is located on the glass at the bottom and the oxidizer droplet (97 wt% H_2O_2) is approaching from above.

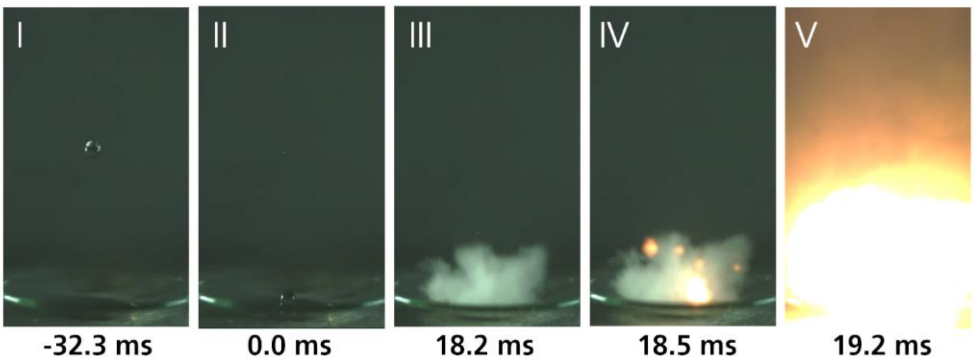

Figure 10. Sequences of a high-speed video of a drop test. Fuel: HIM_35 + 0.05 wt% oGr-NP, Oxidizer: Highly concentrated hydrogen peroxide (97 wt%).

By evaluation of the videos, the ignition delay time is determined. It is defined as the time span between the first contact of oxidizer and fuel (Sequence II) and the first observed flame (Sequence IV) here. Thus, in the example shown

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in Figure 10, the ignition delay time is 18.5 ms. Three drop tests were carried out for each fuel and the average values and standard deviations were determined. A summary of the respective values is given in Table 5.

Table 5: Overview of the average ignition delay times of the nanofluids as fuels with highly concentrated hydrogen peroxide (97 wt%) as oxidizer. Three drop tests were performed for each fuel.

	HIM_35	**HIM_35 + 0.05 wt% oGr-NP**	**HIM_35 + 0.1 wt% oGr-NP**
Ignition delay time Ø [ms]	18.8	18.5	18.9
Standard deviation [ms]	0.14	0.56	0.83

From the values in Table 5 and their graphical representation in Figure 11, it can be derived that the nanoparticles do not affect the hypergolic ignition behavior of the fuels in either a positive or negative way. Especially with regard to the standard deviations, the differences between the various fuel combinations lie in a range in which no influence of the nanoparticles on the hypergolic ignition behavior can be derived.

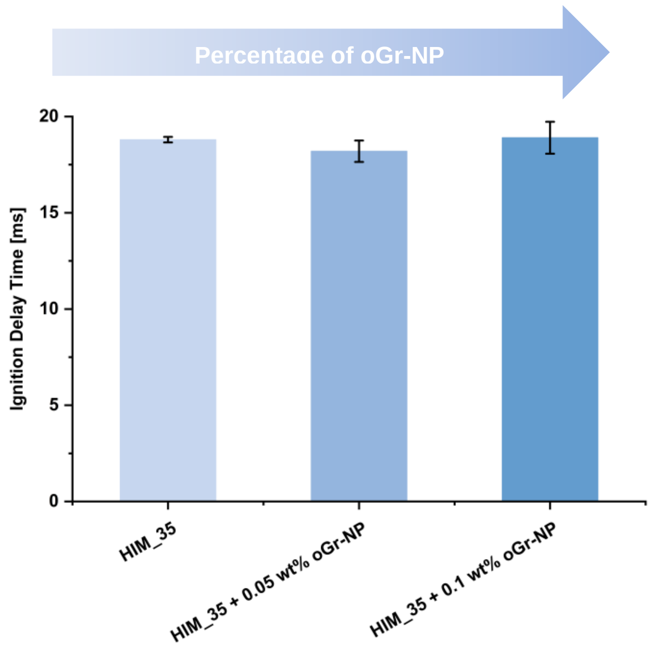

Figure 11: Average ignition delay times with standard deviations of nanofluids as fuels with highly concentrated hydrogen peroxide (97 wt%) as oxidizer.

4. Conclusion and Outlook

Within the DLR intern project *NatAs 2022* (nanoparticles in propulsion systems), carbon nanoparticles were tested as additives in hypergolic ionic liquid fuels and several properties of these fluids were analyzed. Through a comprehensive literature search, six promising carbon-based nanoparticles were selected and extensively characterized in a pre-screening. Some of these were commercially available nanomaterials, while others were synthesized in the laboratory. The size of the nanomaterials was determined by DLS (dynamic light scattering) and SEM (scanning electron microscopy) images. As a result of this screening, polycarboxyl-functionalized graphene nanoplatelets (oGr-NP) at 0.05 wt% and 0.1 wt% were selected for subsequent studies.

In the next step, the ionic liquid-based fuel HIM_35 was defined as the base fluid for the nanoparticle dispersions and the upscaling of the three systems was performed. Characterization of the three test fluids was first performed by density and viscosity measurements. Here, no significant differences were found between the three substances. FTIR spectroscopy was used to assign the characteristic vibrational bands of the samples. Furthermore, TG and DSC analyses

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were performed in the high temperature range up to 600 °C under N_2 atmosphere to determine the influence of the nanoparticles on the decomposition points of the fluids. To characterize the oxidation behavior, these analyses were repeated in the same temperature program under O_2 atmosphere. To characterize the changes of the nanofluids at low temperatures down to -100 °C, TG and DSC analyses were further performed in a low temperature range under He atmosphere. In addition, the suitability of the nanofluids as fuel candidates for hypergolic propellants with hydrogen peroxide (98 wt%) as oxidizer was investigated. For this purpose, the ignition behavior of the propellants was evaluated by hypergolic drop tests on a laboratory scale.

The nanofluids were investigated with regard to their suitability as so-called heat transfer fluids (HTFs). For this purpose, a comprehensive literature search was first carried out and a catalog of requirements was compiled with the aid of HTFs known from literature. In order to classify the test fluids developed here, thermal conductivity, heat capacity and surface tension of the substances were measured experimentally. It was found that the nanoparticles significantly increase the values of thermal conductivity and heat capacity compared to the base fluid HIM_35. Based on this, the follow-up project *NatAs 2023* will focus on further optimizing the properties relevant for HTFs in order to qualify the fuels as possible cooling media in a heat transfer measurement set up. Additionally, the long-term stability of the dispersions will be improved in the following project.

4. Acknowledgement

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