MICROENCAPSULATED HYDROCARBON FUELS IN HYDROGEN PEROXIDE GELS: THE NEXT STEP TOWARD HIGH-PERFORMANCE MONOPROPELLANTS

SPACE PROPULSION 2024
GLASGOW, SCOTLAND | 20 – 23 MAY 2024

Robin Scholl [(1)], Eva Steinmann [(1)], Dominic Freudenmann [(1)] and Stefan Schlechtriem [(1)]

[(1)] German Aerospace Center (DLR), Institute of Space Propulsion, Im Langen Grund, 74239 Hardthausen, Germany Email: robin.scholl@dlr.de

KEYWORDS: green propulsion, gel propellants, hydrogen peroxide, microcapsules

ABSTRACT:

Microencapsulated hydrocarbon fuels in combination with hydrogen peroxide are a new type of monopropellant that unites the high specific impulse of a bipropellant with the efficient hardware of a monopropellant. However, the stabilization of these microcapsule/hydrogen peroxide mixtures is problematic as they tend to separate after a short period of time.

In this work, organic gelling agents are used to prepare stable dispersions of the microcapsule in water and hydrogen peroxide. First, the compatibility of hydrogen peroxide with several gelling agents was investigated and found to be suitable. Next, the dispersion stability of microcapsule/gel mixtures was investigated and found to be stable even at high accelerations in the centrifuge with gelling agent concentrations as low as 0.1%. Together with the subsequent calculation of theoretical performance parameters, these results enable future work on the use of microencapsulated hydrocarbon fuels in combination with hydrogen peroxide as a new monopropellant with the goal of successful hot gas tests.

1. INTRODUCTION

For liquid space propulsion systems usually mono- or bipropellant systems are used [1]. Bipropellant systems typically have a higher specific impulse (I_{sp}) but require more complex hardware such as tanks, pipes and valves, each for oxidizer and fuel. In contrast to that, monopropellant systems have a lower I_{sp} but are easier to build and require less structural material, saving weight and cost [2]. For this, it would be convenient to use hydrogen peroxide as oxidizer component, since it is non-toxic, easy to handle, has a high density and is considered as a green liquid oxidizer [3,4]. The microencapsulation of the fuel in combination with hydrogen peroxide, recently developed in our group, is able to combine the advantages of both mono- and bipropellants, namely a high specific impulse with efficient hardware [3,5,6].

1.1. Implementation of microencapsulated hydrocarbon fuels in propulsion systems

In our previous research, we were already able to show, that the preparation of these hydrocarbon-filled microcapsules is low in cost, easy and reliable: In addition, the compatibility of the polyamide capsule membrane with hydrogen peroxide was demonstrated, with the result that neither the membrane was affected by the hydrogen peroxide nor did the capsules enhance the hydrogen peroxide decomposition rate [3].

The preparation of the hydrocarbon-filled microcapsules is performed by interfacial polymerization with *n*-decane as model substance for a hydrocarbon fuel. For this, a reactive monomer (terephthaloyl chloride, TCL), is dissolved in the hydrocarbon which is then emulsified in water with the help of a surfactant, whereupon a second monomer (diethylenetriamine, DETA) is added to the aqueous phase. Both monomers react at once at the hydrocarbon/water interface and form a polyamide membrane, enveloping the fuel droplets [7]. A depiction of the production process and an optical microscopy image of the resulting microcapsules are shown in Figure 1. The experimental procedure for the synthesis of *n*-decane-filled polyamide microcapsules is shown in section 3.3.

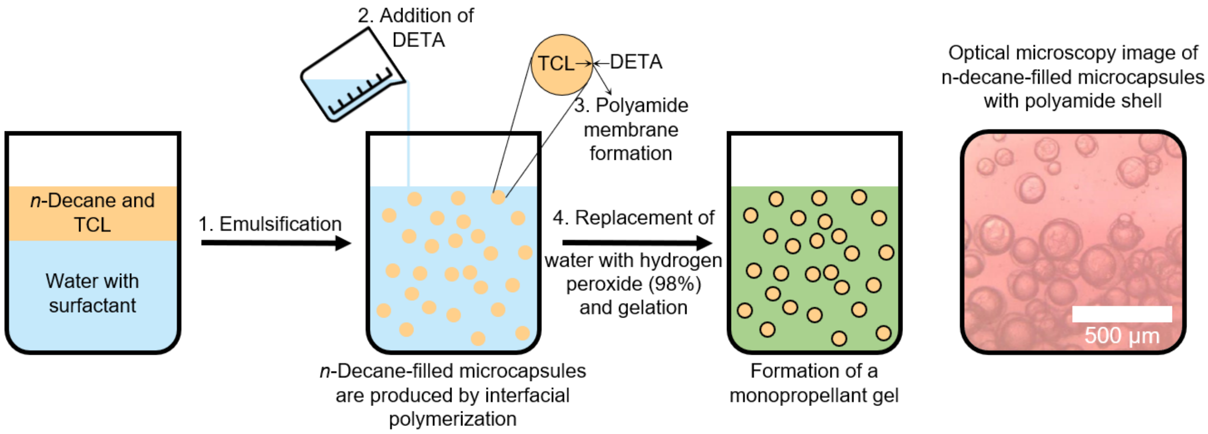

Figure 1. Depiction of the preparation process of n-decane-filled polyamide microcapsules and their formation of a monopropellant with hydrogen peroxide (left) with a gelling agent and optical microscopy images of the capsules in water (right).

1.2. Preparation of stable microcapsule dispersions

A monopropellant that consists of multiple components should be a homogeneous mixture that must be resistant to segregation for long periods of time [4]. Since the density of hydrocarbons is lower than that of hydrogen peroxide (0.7303 g/cm^3 for *n*-decane and 1.4365 g/cm^3 for 98% H_2O_2 at 20 °C [8]), the fuel-filled microcapsules tend to settle on top of the mixture after a short time, a process called creaming. This makes it difficult to use this mixture directly as a monopropellant.

The creaming velocity U can be described by Stoke's law (Eq. 1). It depends on the gravity due to acceleration g, the radius of the particle r, the density of the continuous and dispersed phases ρ_1 and ρ_2 and the viscosity of the continuous phase η_1 [9]. Stoke's law suggests that creaming can be suppressed by different approaches, some of which have already been investigated in previous work by our group [6].

$$U = -\frac{2gr^2(\rho_2 - \rho_1)}{9\eta_1} \qquad \text{Eq. 1}$$

Hereby, water was chosen as a substitute for hydrogen peroxide because it is safe in handling and has similar properties in terms of polarity, melting and boiling point [8].

The first approach was to decrease the size of the microcapsules, since creaming should be slower for smaller capsules. The capsule size can be varied by altering the stirring speed during the emulsification process. However, using microcapsules with a smaller diameter (as small as 4.6 µm on average) creaming could still be observed [6]. Another approach that has been investigated was the use of different surfactants (Span 80, Tween 80, Triton X-100 and polyvinyl pyrrolidine). But neither of those surfactants showed a stabilizing effect on the dispersion [6].

It is therefore necessary to look for other methods to stabilize the dispersion. Promising for this could be a transformation of the continuous phase into a gel by the use of gelling agents.

1.3. Hydrogen peroxide gels

Gels generally consist of a solvent and a gelling agent, which forms a three-dimensional network entrapping the solvent molecules. While the type of interaction responsible for the formation of the network can vary from chemical interactions such as covalent bonding to physical interactions such as entanglement of polymer strands, gels are generally characterized by rheological properties such as shear-thinning behavior and the presence of a yield point. This leads to their classification as non-Newtonian fluids [10].

Gelled propellants in general have been known for some time and are of high interest for chemical propulsion systems since they are able to combine advantages of liquid and solid propellants [11,12]. Propulsion systems using gelled propellants can be throttled and restarted similarly to liquid propellants, but have the ease of handling and storage similar to solid propellants [11]. Gelled fuels also offer the advantage to metallize them for instance with small metal particles, increasing the density and increasing the energetic performance [11,13]. The metallization of fuels has to overcome the same difficulties as in this work, stabilizing small particles in a continuous phase while maintaining good characteristics for the use in a rocket engine.

For hydrocarbon fuels, a large number of gelling agents, both organic and inorganic, are known [14,15]. In contrast, hydrogen peroxide gels are not very well known, despite the fact that hydrogen peroxide has been known and studied in a space propulsion context since the 1930s, either as a green monopropellant or as an oxidizer component in a bipropellant system [1,16].

Most of the known gelling agents for the production of hydrogen peroxide gels concern low concentration hydrogen peroxide in cosmetic and dental applications [17]. However, for gels with 98% hydrogen peroxide, as commonly used in propulsion systems, there are only a few examples described in literature. These include silica [18–20], sodium alginate in combination with calcium cations [21] and cellulose [22]. However, most of these procedures use more than 5% of the gelling agent to achieve successful gelation. In order to affect the combustion properties of the final mixture as little as possible, it would be beneficial to utilize a minimal amount of gelling agent. Additionally, the selected gelling agent should ideally be an organic compound, which can burn without residues [23].

2. RESULTS AND DISCUSSION

2.1. Screening of gelling agents

First, the gelling properties of 24 different compounds were investigated in water as a model substance. Some of these were adapted from literature examples with interest of technical applications of hydrogen peroxide gels, which also used water as a simulant. These included polyacrylic acids (trade name Carbopol (CP)) [20,24,25] and polysaccharide-based agents, such as Xanthan gum [26,27]. An overview of the screened gelling agents is given in Table 4 and the general procedure for the preparation of the gels in section 3.3. The most promising results were achieved with gelling agents based on polyacrylic acid or polysaccharide natural products. In CP/water gels, the rheological properties are best when the pH value is between 5 and 7 [28]. Since CP/water mixtures have a pH value around 3, the pH must be adjusted by adding sodium hydroxide to form a gel. A pH value of 4.5 was identified as optimum to ensure good gelation of the mixture and hydrogen peroxide stability, since hydrogen peroxide tends to a faster decomposition at higher pH values [29,30]. Some of the gels have been rheologically analyzed and they all show shear thinning behavior, a detailed analysis will be part of the future research of our group.

2.2. Gelling agent compatibility with hydrogen peroxide

The gelling agents that delivered the most promising results in the screening tests with water were investigated regarding their effect on the decomposition of hydrogen peroxide. Since hydrogen peroxide decomposition is known to be enhanced by a variety of different compounds [30], the compatibility of hydrogen peroxide with all components of the final mixture has to be evaluated including the influence of sodium hydroxide in the CP/hydrogen peroxide gels. The results are listed in Table 1 and the experimental procedure is shown in section 3.3.

Table 1: Final concentrations observed in the compatibility tests of the gelling agents with hydrogen peroxide after 6 weeks from a starting concentration of 20.2%.

Gelling agent	Hydrogen peroxide concentration after 6 weeks (%)
Reference (pure H_2O_2)	11.6
CP ETD 2691	12.0
Konjac gum	12.0
Xanthan gum	11.6
CP 934 + NaOH	11.5
Tara gum	11.4
CP ETD 2691 + NaOH	11.4
CP 980	11.0
CP 934	10.5
CP 980 + NaOH	10.5
Guar gum	10.3

The results show that while there are some differences in the hydrogen peroxide concentration after the experiment, it is clear that none of the compounds show a strong catalytic effect on the decomposition of hydrogen peroxide. Even the sample containing Guar gum, which had the lowest hydrogen peroxide content, contained only about 1.3% less H_2O_2 than the reference sample. Generally, the hydrogen peroxide content of most samples lies within a range of $\pm$ 0.5% of the reference sample.

No clear effect of the addition of NaOH to the CP gelling agents can be observed. While for CP 980 and CP ETD the final concentration with NaOH was about 0.5% lower than that of the comparative samples without NaOH, this observation could not be confirmed for the sample containing CP 934. Here, the sample with NaOH contains a higher concentration of hydrogen peroxide than the sample without NaOH.

A comparison between the gelling agents based on polyacrylic acids and those based on polysaccharides shows no trends or significant differences between the two substance classes.

2.3. Centrifuge tests

Stoke's law (Eq. 1) states that an increase in g accelerates the creaming process. This can be utilized when trying to test the long-term stability of the dispersions. An experimental setup was conceived that allowed the visualization of the creaming process and the subsequent evaluation of dispersion stability. This concept was based on the fact that the capsules are brighter than the background, and therefore an accumulation of capsules is indicated by an increase in brightness, which is then plotted against the position in the tube. If creaming is occurring, a difference in the distribution of brightness should be visible. The detailed procedure for the centrifuge tests and the analysis of the results is described in section 3.3.

The ratio of oxidizer to fuel (ROF) for a mixture of hydrogen peroxide and the hydrocarbon filled capsules has previously been calculated to be 6.1 at $I_{sp\ max}$ (see section 2.4 and [3]). However, initial tests conducted at this ratio between capsules and gel showed that the concentration of the capsules had to be lowered in order to avoid an oversaturation of the pictures and potentially unreliable results. For this reason, the number of capsules was reduced to an amount equivalent to a ROF of 2.0. The centrifuge tests were conducted with two different capsule sizes, one with an average diameter of 151 µm and smaller ones with a diameter of 24 µm. It is expected that smaller capsules need a lower concentration of the gelling agent to form a stable dispersion, since a decrease in capsule size should increase dispersion stability.

After the procedure was established and verified by centrifugation and comparison of the results to visual observations, it was determined that centrifugation at 50*g* for 60 min is sufficient to claim long-term storability at 1*g*. If the dispersion does not show any signs of creaming and destabilization after that, it can be assumed that the dispersion is probably stable at 1*g* for an extended period of time.

Figure 2 shows an example of the results obtained by the centrifuge tests. The brightness profile and the pictures taken are displayed. It can be seen both in the pictures and in the resulting profiles that no creaming has taken place during the centrifugation, which means the dispersion is considered to be stable. In contrast, Figure 3 shows the result of a stability test with an unstable dispersion. The creaming that occurred during the centrifugation clearly observed in both the brightness profile and the picture taken after 60 min.

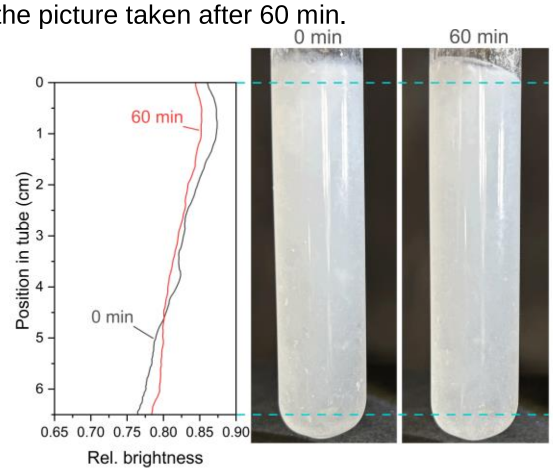

Figure 2: Results of the successful stability testing of a dispersion of 24 µm capsules in 0.1% Konjac gum. Shown is the brightness profile before and after 60 min of centrifugation at 50g as well as the corresponding pictures the profile was extracted from. The blue lines mark the relevant part of the tube that was analyzed.

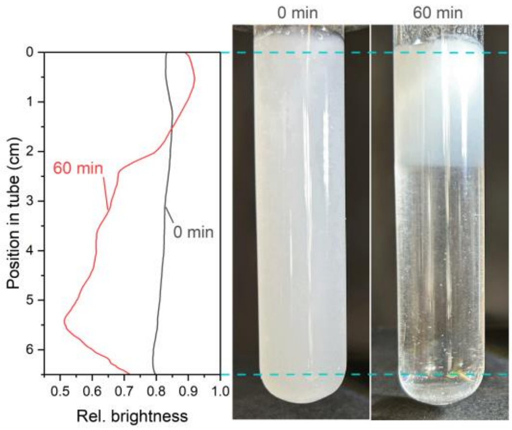

Figure 3: Results of the stability tests of a dispersion of 24 µm capsules in 0.1% CP ETD 2691. Shown is the brightness profile before and after 60 min of centrifugation at 50g as well as the corresponding pictures the profile was extracted from. The blue lines mark the relevant part of the tube that was analyzed.

In order to find the minimal concentration needed to stabilize the dispersion, the centrifugation tests were started with a low concentration, usually 0.1%. If capsule creaming was observed, the gelling agent concentration was gradually increased until a stable dispersion was obtained. In a stable dispersion, the brightness profiles before and after the centrifugation show no sign that creaming has occurred. The results are shown in Table 2.

Table 2: Selected gelling agents and the lowest concentration needed for the capsule dispersion to be stable after centrifugation at 50g for 60 min.

Gelling agent	Gelling agent concentration (151 µm capsules, in %)	Gelling agent concentration (24 µm capsules, in %)
CP 980 + NaOH	0.1	0.2
Konjac gum	0.3	0.1
CP ETD 2691 + NaOH	0.3	0.2
CP 934 + NaOH	0.3	0.2
Xanthan gum	1.0	0.75
Guar gum	> 2.0	1.25
Tara gum	> 2.0	1.75

Generally, the concentration of gelling agent required to obtain a stable dispersion is significantly lower than the values reported previously, where hydrogen peroxide gels typically required a concentration of at least 5% gelling agent, affecting the combustion properties as low as possible. It has also been shown, that the concentration of gelling agent required to stabilize the dispersions depends on the size of the capsules, with the tendency of smaller capsules requiring a lower concentration of gelling agent to be stable.

The investigation of Tara gum and Guar gum with the larger capsules shows that at a gelling agent concentration of 2.0%, some creaming is still visible. Tests at higher gelling agent concentrations were not conducted, as concentrations of 2% are much higher compared to the other gelling agents and lead to the formation of a gel that gets progressively cloudier with increasing gelling agent concentration. However, the evaluation of the brightness profiles works best for transparent gels, and so the results may not be reliable for concentrations above 2.0%.

For this procedure, it has to be taken into account, that the rheological properties of gels such as the phenomenon of a yield stress makes it hard to predict the behavior at 1*g* from observations made at higher accelerations, as would be possible for Newtonian fluids [31]. Therefore, no definitive prediction of the long-term stability of the dispersions can be made from this experiment. Future research will also concern other factors influencing the stability of the final mixture such as hydrogen peroxide decomposition and possible aging effects of the gels. This will also include experiments over prolonged periods of time under the intended storage conditions.

2.4. Performance calculations for gelled capsule-peroxide mixtures

To evaluate the influence of the gelling agent on the performance of the microcapsule/hydrogen peroxide monopropellant, theoretical calculations were conducted using NASA CEA with polyacrylic acid as a model gelling agent. The results are listed in Table 3. The maximum I_{sp} of the microcapsule/hydrogen peroxide mixture (336.7 s) significantly exceeds the performance of established monopropellants, such as SHP 163 (I_{sp} = 276 s [3,32]), with a comparable density. The $I_{sp\,max}$ of the gelled mixtures is only slightly reduced by the quantities of gelling agents that were used in the experiments (334.6 s with 1.0% of polyacrylic acid as gelling agent), while the ROF remains at 6.1 throughout this concentration range.

Table 3. Calculation of the theoretical performance of gelled microcapsule/hydrogen peroxide monopropellants in dependence of the amount of gelling agent (polyacrylic acid).

Amount polyacrylic acid (%)[a]	$I_{sp\,max}$ (s)[b]	ROF at $I_{sp\,max}$[b]
0	336.7	6.1
0.1	336.5	6.1
0.3	336.1	6.1
1.0	334.6	6.1

[a]Weight amount in respect to hydrogen peroxide mass [b]Calculated combustion data using NASA CEA (assuming frozen supersonic expansion, ε = 330, p_c = 10 bar) using thermodynamical data from section 3.2 and literature [8,33].

3. EXPERIMENTAL

3.1. General information

All solvents and reagents were purchased from Acros Organics (polyvinyl alcohol 88% hydrolyzed (MW 20 000- 30 000)), Merck (*n*-decane), Sigma Aldrich (diethylenetriamine) and Thermo Scientific (terephthaloyl chloride) and were used without further purification. A Heidolph RZR 2020 with dispersion blade and an IKA T18 digital ULTRA TURRAX with a S 18 N – 19 G dispersion tool were used as dispersers

Table 4: List of screened gelling agents and their suppliers.

Gelling agent	Supplier
Carbopol 980	Serva
Carbopol 934	Caesar
Carbopol ETD 2961	Lubrizol
Aculyn	The Dow Chemical Company
Aerosil 200	Evonik
Aerosil R972	Evonik
Cellosize QP-52000H	The Dow Chemical Company
EKAGUM LR1	Textilchemie Dr. Petry GmbH
EKAGUM LR2	Textilchemie Dr. Petry GmbH
EKAGUM LR3	Textilchemie Dr. Petry GmbH
Galactomannan	Carl Roth
Gelatin	Acros Organics
Guar gum 5000 cps	TER Chemicals
Guar gum L. Taub	Naturkosmetik-Werkstatt
Karaya gum	Alfa Aesar
Konjac gum	TER Chemicals
Locust bean gum	TER Chemicals
PNC 400	Naturkosmetik-Werkstatt
Polyvinylpyrrolidon K90	Carl Roth
Sodium Alginate	Carl Roth
TAFIGEL PUR 61	Münzing Chemie GmbH
Tara gum	TER Chemicals
Tragacanth	Alfa Aesar
Xanthan gum	Kosmetische Rohstoffe

3.2. Analytical methods

Centrifugation was conducted with a Heraeus Biofuge primo. The value of the acceleration is given in *g* with 1*g* = 9.81 m/s^2.

Concentration determination of hydrogen peroxide The concentration of hydrogen peroxide was determined by density measurement with a Mettler Toledo Easy D40 at 25 °C and the use of literature data to convert the density to a weight percentage value [34]. The measurement uncertainty is ±0.0005 g/cm^3.

All concentration values (hydrogen peroxide and gelling agents) are given in percentage of weight.

Heat of combustion was measured with an IKA C 200 auto. The heat of combustion for polyacrylic acid (Carbopol ETD) was measured three times (*M* = 17.01 kJ/g, *SD* = 0.06 kJ/g).

Optical microscopy Images were obtained by a Leica DM2700 M optical microscope.
Rheology The rheological properties of the gels were measured with a Haake RheoStress 6000 rotational rheometer with a cone-plate geometry (35 mm diameter plate and 2° cone) at 20 °C.

3.3. Experimental procedures

Preparation of *n*-decane filled diethylene-triamine – terephthaloyl chloride microcapsules [3,6]
Polyvinyl alcohol (150 mg) was diluted in water (30 mL) and a solution of terephthaloyl chloride (131 mg, 643 µmol, 1.0 equiv.) in *n*-decane (5 mL) was added. The mixture was then dispersed for 5 min. Consequently, a solution of diethylene-triamine (2.07 mL, 19.3 mmol, 30.0 equiv.) was added and the solution was gently stirred overnight. Subsequently, the resulting capsules were washed with water (4 × 100 mL).

General Procedure for preparation of the gels
While stirring, 0.2 g of the gelling agent were added to 20 mL of water. The mixture was stirred for 5 min at 650 rpm and subsequently observed over a period of 24 hours. Whenever necessary and indicated in the technical datasheets of the compound or previous literature, the reaction conditions were modified by adjustments of the pH value or heating during the process.

Compatibility testing with hydrogen peroxide
10 g of 20.2% hydrogen peroxide and 0.1 g of the gelling agent were mixed with a glass rod. The samples were stored in glass containers with the lid placed lightly on top to allow for the release of oxygen gas, which is formed during the decomposition of hydrogen peroxide [30].
Those glass containers were stored in the dark at room temperature and the changes in weight of the samples were observed over a period of 6 weeks. It was assumed, that the weight loss is caused by the loss of oxygen gas released by the decomposition of hydrogen peroxide. This is then transferred to a concentration value and compared to a sample of pure hydrogen peroxide kept in the same conditions.

Preparation and evaluation of centrifuge tests
While stirring, the desired quantity of gelling agent was added to room temperature water (30 mL). The mixtures were stirred for 30 min and then left to stand for another 30 min. Then the microcapsules (0.5 g; wet) were added into a clear and unmarked centrifuge tube and 10 mL of the gel was added. In the case of the Carbopol gels, a 10% NaOH-solution was added to adjust the pH value to 4.5. The mixtures were stirred with a glass rod until the capsules were evenly dispersed and left to rest for 60 min. The tubes were photographed, centrifuged at 50*g* for 60 min and photographed again. The pictures were taken with an iPhone 13 and the relevant part of the image, containing the centrifuge tube and its contents, was cropped out. Using MATLAB this was then converted to greyscale, in which the value of each pixel is a representation of brightness. The mean value of each row of pixels was then calculated and applied against the position in the tube. The brightness values were converted to relative values on a scale between 0 (minimum brightness, black) and 1 (maximum brightness, white). The curves were smoothed with the Savitzky-Golay method.

4. CONCLUSION

In this work, a crucial step in the development of a new type of monopropellant that consists of fuel-filled microcapsules dispersed in hydrogen peroxide was taken by stabilizing these dispersions with organic gelling agents. For this, several gelling agents were screened for their gelling properties using water as a model substance. The most promising candidates were then selected for compatibility tests with hydrogen peroxide and showed no evidence of potential incompatibility. It has also been shown that the microcapsule/gel dispersions are stable over a prolonged period of time through the use of a centrifuge. Extremely low concentrations of gelling agent, as low as 0.1%, can result in stable dispersions. In addition, calculations of the theoretical performance of these gels were performed, demonstrating their high potential as a powerful monopropellant.

Together, these results allow for future work using microencapsulated hydrocarbon fuels combined with hydrogen peroxide as a new monopropellant with the goal of successful hot gas tests.

CRediT authorship contribution statement

Robin Scholl: Conceptualization, Investigation, Writing – original draft.

Eva Steinmann: Investigation, Writing – original draft.

Dominic Freudenmann: Funding acquisition, Resources, Writing – review & editing.

Stefan Schlechtriem: Supervision.

Acknowledgements
The authors would like to thank the team members of the Department for Chemical Propellant Technology at the DLR Institute of Space Propulsion for their support.

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