

Safety and Performance Evaluation of a New High-Performance Monopropellant Based on a Microencapsulated Hydrocarbon Fuel in Combination with Hydrogen Peroxide using a Strand Burner Setup

*Robin Scholl *†, Felicitas S. Moll *†, Dominic Freudenmann * and Stefan Schlechtriem **

** German Aerospace Center (DLR), Institute of Space Propulsion
Im Langen Grund, 74239 Hardthausen, Germany*

† Corresponding Authors: robin.scholl@dlr.de; felicitas.moll@dlr.de

Abstract

Conventional monopropellants only offer a limited specific impulse and often exhibit toxic properties. Therefore, our group developed a new microencapsulated hydrocarbon monopropellant combined with hydrogen peroxide, offering a non-toxic, environmentally friendly alternative with a high specific impulse. This study presents the results of the first combustion tests of this new monopropellant, which were conducted using a strand burner setup to investigate the influence of different encapsulated fuels on the burn rate. Additionally, BAM Fallhammer tests were conducted to evaluate the impact sensitivity of the monopropellant in relation to capsule size and burn rate, in order to assess its safety characteristics.

1. Introduction

Liquid rocket propellants can be categorized as either bipropellants or monopropellants. [1] Bipropellants consist of fuel and oxidizer components that are stored separately and can only react with each other in the combustion chamber. This separation enables a high degree of controllability as well as energetically advantageous combinations, which is reflected in high specific impulses. However, bipropellants are also associated with high technical requirements, significantly increasing factors such as weight and system complexity. Monopropellants, on the other hand, consist of a single, reactive liquid or a homogeneously mixed component which reacts through catalysis or thermal ignition. While these systems enable significantly easier handling and greater reliability, they usually come at the cost of a lower specific impulse. [2]

A central problem with many conventional propellants is their high toxicity and environmental impact. Substances such as hydrazine, 1,1-dimethylhydrazine (UDMH) and nitrogen tetroxide (N_2O_4), which have long been used as standard propellants, are considered to be highly toxic, carcinogenic and harmful to the environment. The handling of these substances also requires extensive protective measures, making their use increasingly problematic. [3, 4] In recent years, so-called “green” propellants have therefore been increasingly developed, including ionic liquids based on UAN (urea-ammonium nitrate) or ADN (ammonium dinitramide), which have lower toxicological risks. [5, 6] Despite this progress, green monopropellants still have significantly lower specific impulses than conventional bipropellants. [7] So-called premixed monopropellants, in which oxidizer and fuel are homogeneously mixed prior to the reaction, have also emerged as a focus of interest. While these systems enable compact storage and potentially simplified ignition, they also raise safety concerns especially blends of hydrogen peroxide as oxidizer with organic components such as alcohols. [8, 9]

In this context, our research group has developed a novel monopropellant system combining hydrogen peroxide (H_2O_2) as an oxidizer with microencapsulated hydrocarbon fuel. [10, 11] By physically separating the two components in a stable suspension, the reactivity of the two substances compared to unprotected mixed organic components with hydrogen peroxide can be reduced. Initial thermochemical analyses indicate a specific impulse of about 330 s, placing it in the performance region of classic bipropellant systems. Furthermore, it has been experimentally proven that the components remain stable in the suspension over a prolonged period of time and do not have a negative influence on each other, which is a fundamental prerequisite for potential applications. [10]

This work aims to systematically investigate the combustion behavior of the newly developed monopropellant for the first time. Ignition and combustion behavior tests are carried out in a specially constructed strand burner setup. The influence of different encapsulated hydrocarbon-based fuels on the combustion, as well as the influence of the capsule size on the impact sensitivity, were analyzed. The knowledge gained can be used for detailed characterization, optimization and safety-related evaluation of the propellant system in preparation for possible hot-fire tests.

2. Experimental Section

2.1 Chemicals

All commercially available solvents and reagents were used without further purification. The reagents were purchased from Acros Organics (polyvinyl alcohol 88% hydrolyzed (MW 20 000 - 30 000)), Merck (*n*-pentane), Alfa Aesar (*n*-heptane), Merck (*n*-decane), Acros Organics (*n*-dodecane), Merck (*n*-pentadecane), Sigma Aldrich (diethylenetriamine), Thermo Scientific (terephthaloyl chloride), Lubrizol (Carbopol ETD 2961).

2.1 Analytical methods

Optical microscopy Images have been taken using a Leica DM2700 M optical microscope.

Centrifugation was conducted with a Sigma 3-18KS centrifuge. The value of the acceleration is given in g with $1g = 9.81 \text{ m/s}^2$.

Dispersions were prepared using a Heidolph RZR 2020 with an IKA R 1303 dissolver stirrer and an IKA T18 digital ULTRA TURRAX with a S 18 N – 19 G dispersion tool.

Mechanical sensitivity Impact sensitivity was tested using a BAM Fallhammer BFH-10 manufactured by OZM Research following the 1-in-6 method. [12]

Concentration determination of hydrogen peroxide (in percentage of weight) 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 [13]. The measurement uncertainty is $\pm 0.0005 \text{ g/cm}^3$.

Highspeed videos were filmed by a Photron fastcam SA-X2 color high-speed camera.

2.1 Experimental procedures

Preparation of *n*-alkane filled diethylene triamine – terephthaloyl chloride microcapsules [14]

Polyvinyl alcohol (150 mg) was diluted in water (30 mL) and a solution of terephthaloyl chloride (83.7 mg, 412 μmol , 1.0 equiv.) in *n*-alkane (5 mL) was added. The mixture was dispersed for 5 min. Consequently, a solution of diethylenetriamine (2.07 mL, 19.3 mmol, 30.0 equiv.) in water (10 mL) was added and the mixture was gently stirred overnight. The resulting capsules were separated in a separatory funnel or by centrifugation (1 to 107 g) and washed with water (4 x 100 mL). The average size of the microcapsules was determined using an optical microscope (based on the measurement of 50 capsules).

Preparation of the hydrogen peroxide gel [11]

The gelling agent Carbopol ETD 2961 (60 mg) was added to hydrogen peroxide (20 g) under vigorous stirring at 650 rpm for 5 min. Afterwards, an aqueous sodium hydroxide solution (120 μL , 10% NaOH) was added and the mixture was stirred for additional 5 min.

Preparation of the microencapsulated propellant

For preparation of the propellant, the microcapsules were separated by centrifugation (107 g) and washed with hydrogen peroxide (3 x 20 mL). The previously prepared hydrogen peroxide gel was then added to the capsules according to the required mass fraction in order to obtain the desired ROF. The combustion data of the *n*-alkane filled microcapsules were calculated using NASA CEA (assuming frozen supersonic expansion, $\varepsilon = 330$, $p_c = 10 \text{ bar}$) using thermodynamical data from [15].

General Procedure for determining the burn rate

For analysing the burn rate, a strand burner setup consisting of a stainless steel chamber was used (compare to Figure 1). A tube holder for inserting a borosilicate tube is located in the middle of the chamber. On the side of the tube holder are two phenolic posts with a crocodile clip to which the ignition wire is attached. In order to be able to pressurize the chamber with an inert gas, for the experiments at elevated pressure, the chamber is equipped with sapphire glass windows at the front and rear, sealed off with two graphite gaskets. On the side of the chamber there are several access ports to which pressure gauges, pressure relief valves and the gas inlet and outlet is attached.

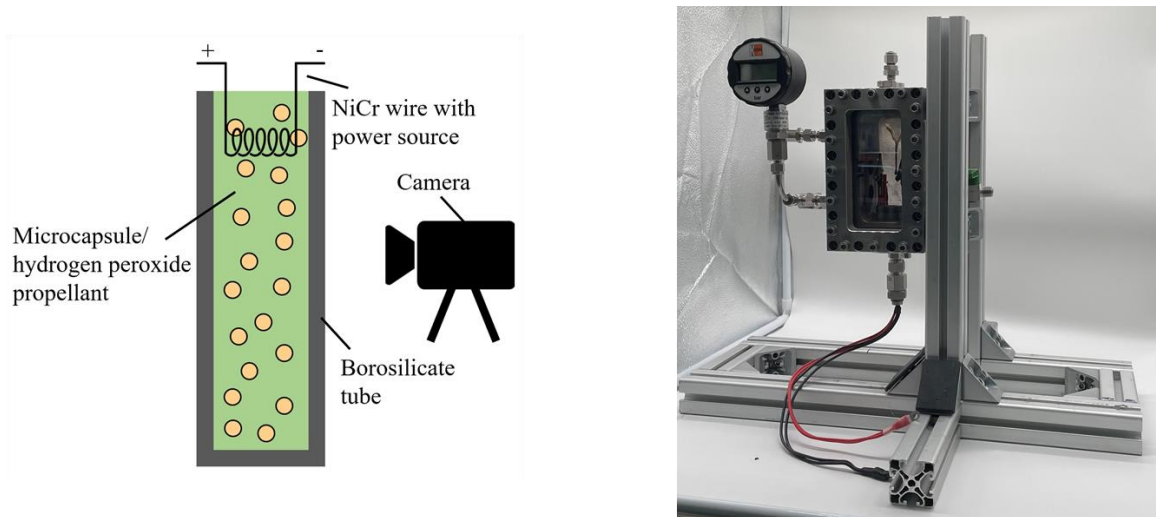


Figure 1: Schematic illustration of the strand burner setup used in this work (left) and image of the experimental setup (right).

The propellant is filled into the borosilicate tube (outer diameter = 9 mm, inner diameter = 6 mm, length = 70 mm) and placed into the tube mount. The propellant is ignited by a coiled Nichrome (NiCr) wire (87 Ω /m, 0.4 mm diameter, 80% nickel, 20% chromium), which is heated by resistive heating by passing an electric current through it. The propagation of the flame is recorded by a camera and the burn rate is later analysed visually by measuring the time it takes the flame to travel through a distance of 3.5 cm (markings on the tube). An example of a combustion test is shown in Figure 4, the 3.5 cm distance is covered in 32 s, which corresponds to a burn rate of 1.09 mm/s. We found no evidence of significant variation in the burn rate during a single test. Each test was repeated 3 times for each measurement point to ensure reproducibility.

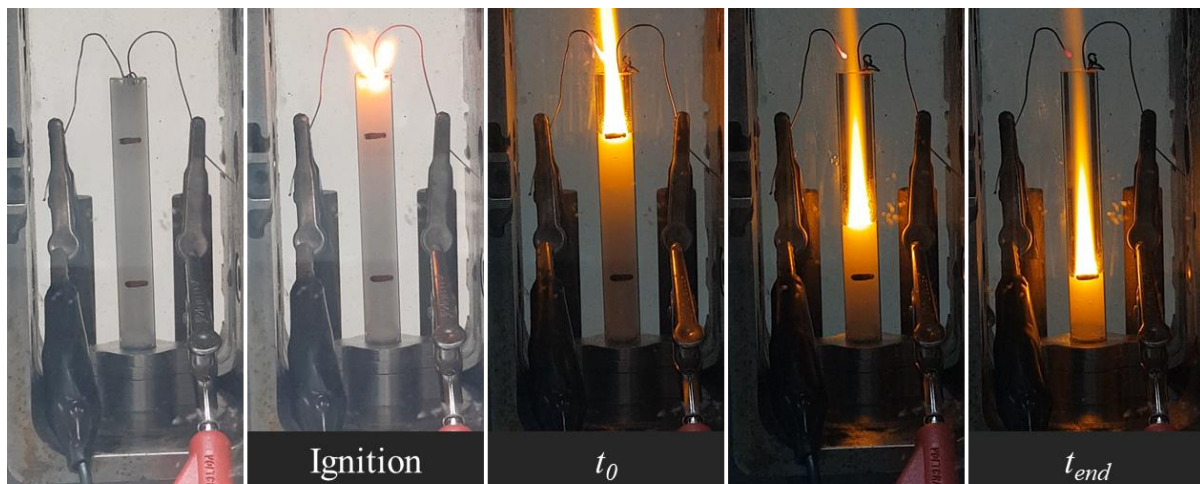


Figure 2: General procedure of a combustion test, the test parameters were: average capsule size: 24.1 μ m, hydrogen peroxide concentration: 90%, ROF: 7, fuel: *n*-decane, pressure: 1013 hPa.

3. Results and Discussion

To characterize the novel monopropellant, various parameters were systematically changed and their influence on the burn rate was investigated. A particular focus was placed on the fuel used within the polyamide capsules and the capsule size. Other parameters, such as the hydrogen peroxide concentration, the ratio of oxidizer to fuel (ROF), the system pressure during the combustion tests and the concentration of the gelling agent, were kept constant during the test series to ensure the comparability of the results. Each individual test was repeated three times for each measuring point, with the resulting inaccuracies displayed in the error bars. A gelling agent concentration of 0.3 wt%, an ambient pressure of 1013 hPa, an ROF value of 7 and a hydrogen peroxide concentration of 90% were defined as standard conditions for the test series.

As the investigated system is a high-energy monopropellant, safety aspects were also considered. For this purpose, BAM Fallhammer tests were carried out to evaluate the impact sensitivity behavior of the system. In particular the influence of the capsule size on the impact energy and the subsequent thermal behavior was investigated. Figure 1 shows an image of the microcapsules taken with an optical microscope.

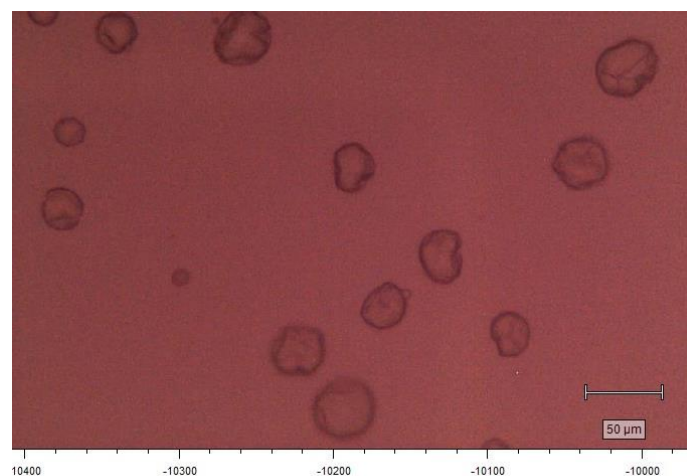


Figure 3: Optical microscopy image of *n*-dodecane filled microcapsules.

3.1 Fuel variation

To investigate the influence of different fuels within the polyamide capsules on the combustion behavior, propellants with *n*-alkane-filled microcapsules with different chain lengths were prepared respectively and examined in the strand burner setup. For better visualization, the *n*-alkanes were arranged according to their boiling temperatures. The corresponding findings are shown in Figure 4.

It is apparent that the burn rate increases with increasing chain length of linear *n*-alkanes, and appears to reach its highest with dodecane and seems to plateau towards even longer chains (pentadecane). The observed empirical trend is likely to be explained by the interaction of various factors, such as boiling temperature, enthalpy of vaporization and ignition temperature. These factors may not act independently, but could influence each other to some extent. However, in order to be able to make well-founded conclusions about which specific material properties are responsible for changes in the burn rate, and the extend to which they contribute individually, further investigations and an extended dataset are required.

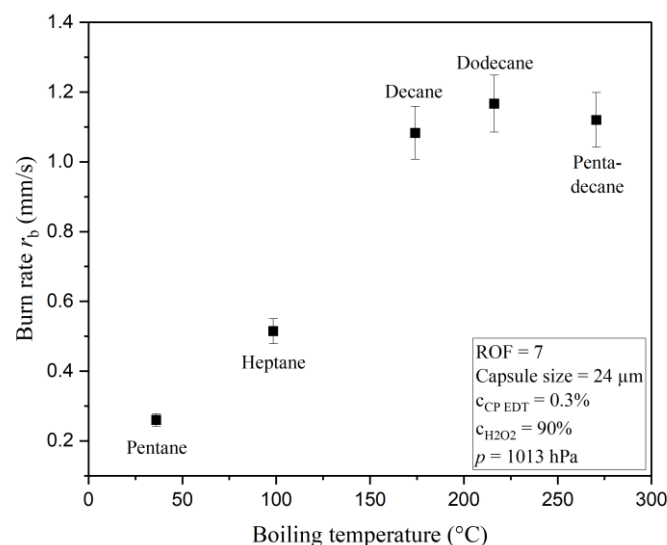


Figure 4: Average burn rate of *n*-alkane filled microcapsules in dependence of the boiling temperature [15].

3.2 Thermal decomposition and safety assessment with BAM Fallhammer

Another interesting behavior, especially in systems with higher burn rate, was the occasional occurrence of spontaneous thermal decomposition of the propellant during the ignition process in the strand burner. In order to selectively investigate this behavior, experiments were carried out in which thermal decomposition was to be induced by a high thermal energy input. This energy input depends largely on the contact area between the ignition wire and the propellant as well as on the current and therefore the temperature of the ignition wire. Figure 5 shows individual frames of a high-speed video, recorded at 150 000 frames per second, which document the course of the thermal decomposition. The total duration of this event is less than 0.1 ms.

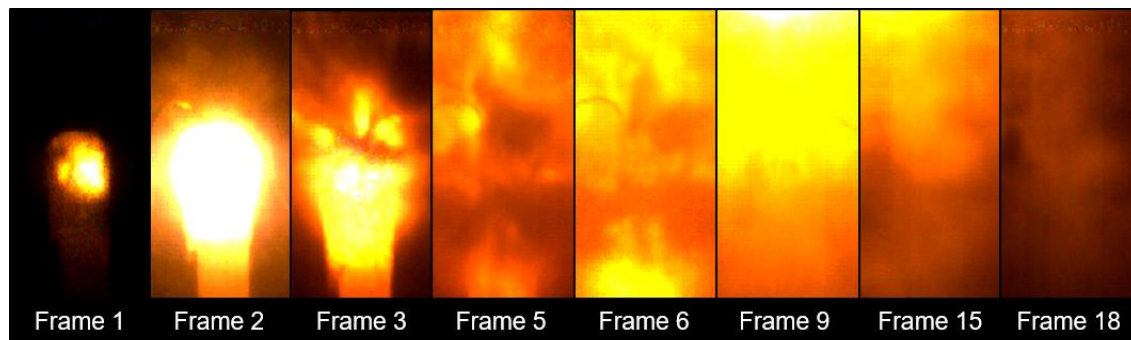


Figure 5 Single high-speed video frames of the spontaneous thermal decomposition, the individual frames are numbered in the picture (150 kfps and an exposure time of 1/177049 s). Sample parameters: average capsule size: 4.6 μm , hydrogen peroxide concentration: 90%, ROF: 7, fuel: *n*-decane.

In order to gain a better understanding of this reactive behavior of the propellant, the impact sensitivity of the system was investigated by varying selected parameters. The focus was particularly on the influence of the capsule size onto the impact sensitivity. For this purpose, *n*-decane filled microcapsules with different diameters were prepared. The average capsule size of the *n*-alkane filled microcapsules was adjusted by modifying the stirring speed during the dispersion process, whereby higher stirring speeds lead to a decrease in capsule size. [14] The minimal impact energy of the corresponding propellants was then determined by BAM Fallhammer tests.

It was found that the lowest impact energy increases steadily with increasing capsule size, following a root-shaped curve and finally reaches a plateau, which is shown in Figure 6. This behavior indicates that the reactivity of the system increases with the use of smaller capsule sizes. This increase in reactivity is accompanied by an increasing burn rate of the propellants with decreasing capsule size, as already shown in previous work of our group. [16] It can probably

be concluded that smaller microcapsules lead to a more homogeneous distribution of the fuel within the hydrogen peroxide gel. This more uniform distribution results in a narrower spread of local maxima and minima within the propellant, enabling it to react optimally without being too thick or too lean in any one area. Additionally, the higher total surface area of smaller capsules can generally lead to higher reaction rates.

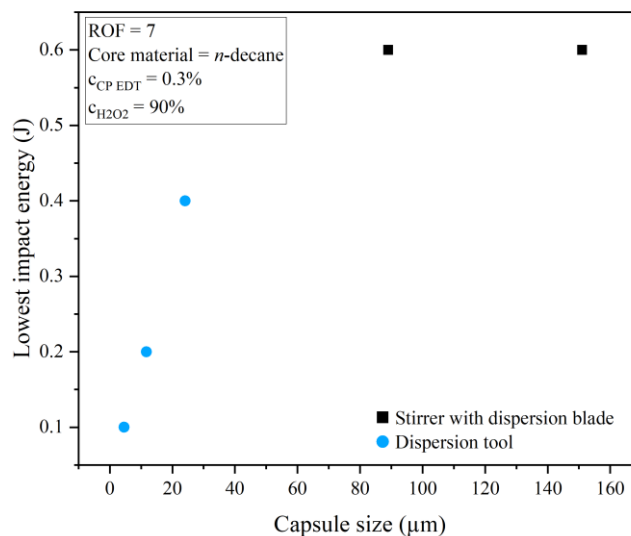


Figure 6: Lowest impact energy of the microcapsule propellant in dependence of the capsule size determined with a BAM Fallhammer by the 1-in-6 method [12].

4. Conclusion and Outlook

The results clearly demonstrate that the fuel contained within the microcapsules has a significant influence on the burning rate of the propellant. This finding provides a valuable basis for further investigations and for targeted propellant adaption, particularly with regard to hot fire tests. Furthermore, the range of possible customizations could be expanded by analyzing various fuels. This would enable the burning behavior to be specifically adapted to suit individual operating conditions.

Additionally, it was found that the capsule size is directly correlated with the system reactivity. This is particularly relevant when it comes to handling, storing or assessing the safety of the propellant. As this system is highly energetic, a thorough safety evaluation is essential to ensure responsible and low-risk handling. To gain a better understanding of the correlation between burn rate and lowest impact energy, future research should investigate whether this correlation applies exclusively to the capsule size variation, or if it can be transferred to other parameters. To this end, the impact sensitivity and related burn rate of other influencing variables could be compared. The aim would be to identify and validate potential correlations in order to gain a more comprehensive understanding of the system behavior.

CRedit authorship contribution statement

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

Felicitas S. Moll: Investigation, Writing – original draft.

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

Stefan Schlechtriem: Supervision.

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