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Performance Evaluation of a High-Performance Monopropellant Formed of Microencapsulated Hydrocarbon Fuels in Combination with Hydrogen Peroxide in a Strand Burner Setup

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Abstract
A new type of monopropellant, consisting of fuel-filled microcapsules in combination with hydrogen peroxide as oxidizer, combining the high specific impulse of a bipropellant with the efficient hardware of a monopropellant, has been developed by our group over the past few years. In this work, a first study on the combustion behavior of this propellant was performed, using a strand burner setup. The influence of different propellant parameters (oxidizer to fuel ratio, capsule diameter, hydrogen peroxide concentration and pressure dependency) on the burn rate was evaluated, in order to further develop this system for future hot-fire tests.

1. Introduction

In liquid propulsion systems, usually mono- or bipropellants are used [1]. Monopropellant systems generally offer the advantage of a high reliability and a reduced number of components, saving weight and cost. However, they typically compromise a lower specific impulse compared to bipropellant systems. The development of monopropellants with a higher specific impulse is therefore of significant interest, bridging the performance gap while maintaining simplicity [2].

Considering conventional liquid "green" monopropellants optimized for a high performance, they often consist of an energetic ionic liquid such as hydroxylammonium nitrate (HAN) or ammonium dinitramide (ADN) as the oxidizing component together with some type of fuel. Hereby a specific impulse (I_{sp}) of around 260-280 s can be achieved [3]. But there is still a significant gap to typical bipropellant systems like hydrazine and its derivatives in combination with nitrogen tetroxide or hydrogen peroxide in combination with hydrocarbon fuels which usually have an I_{sp} of 320 s and above [4]. Focusing on "green" propellants, hydrogen peroxide really stands out as oxidizing component since its non-toxic, easy to handle and well studied [5]. In the past, several attempts have been made to develop a hydrogen peroxide-based monopropellant with a high specific impulse by mixing it with polar organic fuels. Recent examples include the work of Kwon et al. who investigated blends of hydrogen peroxide with ethanol or tetraglyme [6,7]. However, these premixes inherently carry a significant risk of spontaneous decomposition due to their high energy content, raising serious safety concerns even at lower concentrations of hydrogen peroxide [8,9].

One possible solution for creating a safe high-performance premixed monopropellant based on hydrogen peroxide is to blend it with a microencapsulated hydrocarbon fuel. The encapsulation keeps the fuel and oxidizer separate and allows them to be safely stored together in one tank. This concept has been developed by our group over the past few years [10,11]. Hereby we were already able to show, that the preparation of these hydrocarbon-filled microcapsules and their combination with hydrogen peroxide is low in cost, easy and reliable. The produced propellant has a high density (1.27 g/cm^3) and an I_{sp} of up to 330 s [11,12]. In addition, the compatibility of the polyamide shell material 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 [11].

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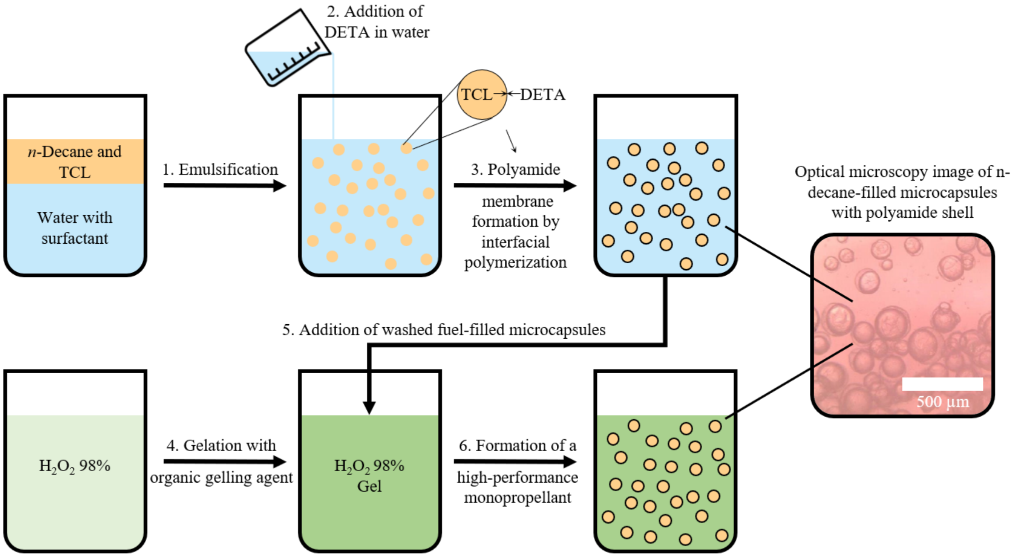

Figure 1: Preparation of the fuel-filled microcapsules, synthesized by an interfacial polymerization (top) which are then dispersed in a hydrogen peroxide gel to form the propellant (bottom); an optical microscopy image of the resulting microcapsules is shown on the right.

In Figure 1, the preparation process of the microencapsulated propellant is shown. For synthesis of the microcapsules, a reactive monomer (terephthaloyl chloride, TCL) is dissolved in a hydrocarbon fuel like *n*-decane, which is then emulsified in water with the help of a surfactant. Subsequent addition of a second monomer (diethylenetriamine, DETA) to the aqueous phase leads to an immediate reaction of both monomers at the hydrocarbon/water interface. Hereby a crosslinked polyamide membrane is formed around the droplets, enveloping the fuel [13]. Meanwhile a hydrogen peroxide gel is prepared with a polyacrylic acid-based gelling agent, into which the microcapsules are then mixed to form the high-performance monopropellant. The gelling agent is required to prevent the capsules from settling on top of the mixture (a process called "creaming") [12,14]. The detailed experimental procedure is outlined in section 2.3.

1.1 Strand burner setup

Strand burners have long been the standard method for evaluating the burn rate of propellants under controlled conditions. This data is critical to the further development of the propellant to ensure stable, safe and efficient combustion and to provide a fundamental set of data for the design of a suitable engine and further testing. [15]
Traditionally applied to solid propellants, a cylindrical strand of the propellant is ignited and the burn rate is determined by tracking the regression of the flame front over time. The system can also be pressurized, simulating the conditions inside a combustion chamber, to determine the pressure dependency of the burn rate. These devices also allow the measurement of the burn rates for liquid or gelled propellants [16,17]. In Figure 2, a schematic depiction of the strand burner setup used in this work is shown, the propellant is filled in a borosilicate tube and ignited at the top with a NiCr wire and the progress of the flame front is tracked with a camera. More details on the experimental procedure and a picture of the actual setup is given in section 2.3.

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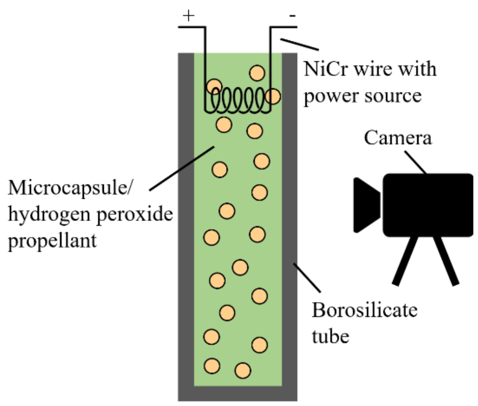

Figure 2: Schematic depiction of the strand burner setup used in this work.

In this work, we present a first set of combustion experiments and burn rate determinations of this new microcapsule/hydrogen peroxide propellant. These include variations in the mass ratio of oxidizer to fuel (ROF), capsule size, hydrogen peroxide concentration and experiments at elevated pressures.

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*-decane), Sigma Aldrich (diethylenetriamine), Thermo Scientific (terephthaloyl chloride) and Lubrizol (Carbopol ETD 2961)

2.2 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 1*g* = 9.81 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.

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 [18]. The measurement uncertainty is ±0.0005 g/cm^3.

2.3 Experimental procedures

Preparation of *n*-decane filled diethylene triamine – terephthaloyl chloride microcapsules [11]
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*-decane (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 [12]
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.

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Preparation of the microencapsulated propellant

For preparation of the propellant, the microcapsules were separated by centrifugation (107g) 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.

General Procedure for determining the burn rate

For analyzing the burn rate, a strand burner setup was used (compare to Figure 3). This consists of a stainless steel chamber with saphire glass windows in the front and the rear, sealed with two graphite gaskets. 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 isolated posts with a crocodile clip to which the ignition wire is attached. Argon (5.0 purity) was used to pressurize the chamber. The pressure during the combustion process was controlled using an overflow valve for each pressure step.

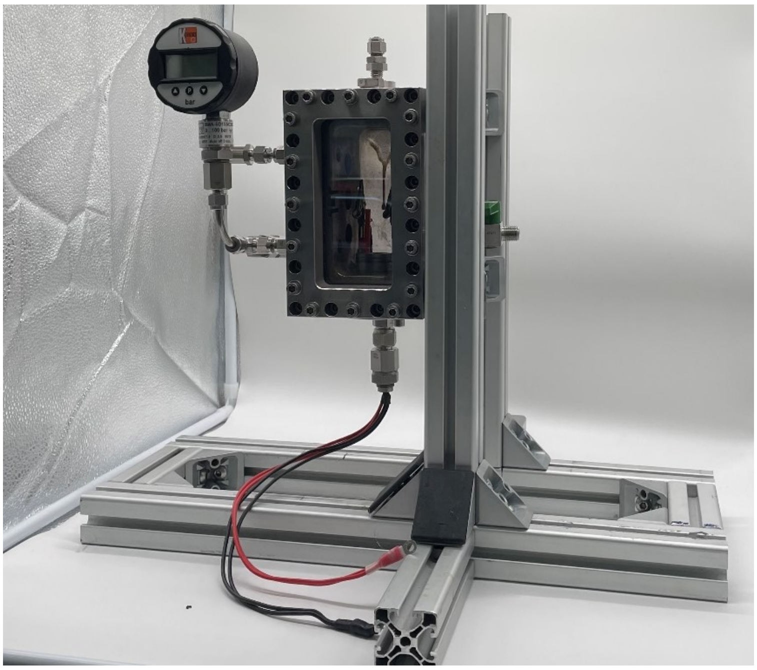

Figure 3: Image of the experimental setup of the strand burner.

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 analyzed 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. Here, 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.

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Figure 4: Exemplary frames of the video from a combustion test, the combustion rate is determined by measuring the time span (from t_0 to t_{end}), the flame front needs to travel a defined length (3.5 cm) in the glass tube.

3. Results and discussion

Several parameters were varied in the combustion tests in order to observe their influence on the burn rate. This involved investigating the influence of changes in the ROF, the hydrogen peroxide concentration, the average capsule size and the pressure dependency. In order to enable comparability of the results, the other parameters were always kept constant within a test series. Each combustion test was conducted three times for one measurement point and the standard deviations are given in the error bars. A capsule size of 24.1 µm, a gelling agent concentration of 0.3 wt% and a hydrogen peroxide concentration of 90% with an ROF value of 7 are considered as the standard conditions.

3.1 ROF variation

Calculations showed, that the $I_{sp\ max}$ of the microcapsule/hydrogen peroxide (98%) propellant is at a ROF of 6.1 [11]. To show that the optimum ROF for the combustion tests is at the maximum calculated specific impulse I_{sp}, several combustion tests were carried out with varying ROFs. The relationship between the burn rate and the selected ROFs can be seen in Figure 5. The results show, that the optimum ROF for the ignition tests corresponds to the calculated ROF at $I_{sp\ max}$. The ROF at the calculated $I_{sp\ max}$ is therefore always used in the subsequent tests.
It is interesting to note that both the specific impulse and the burn rate show a similar strong increase in number at lower ROFs and both reach their maximum at a ROF of approximately six. At higher ROF values however, there is a divergent behavior to be seen: the burn rate decreases much faster than the specific impulse.

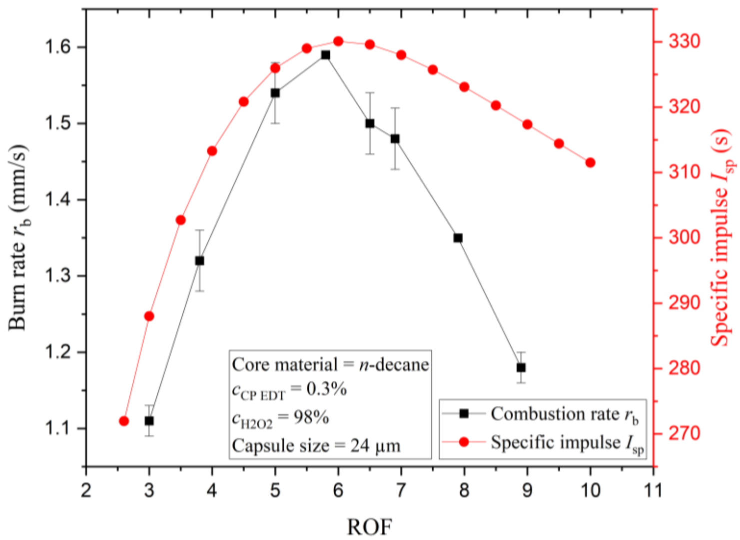

Figure 5: Average burn rate of n-decane filled microcapsules (average size: 24.1 µm, hydrogen peroxide concentration: 98%) in dependence of the ROF and the theoretically calculated specific impulse in dependence of the ROF.

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3.2 Variation of the capsule size

In order to investigate the influence of capsule size on the combustion rate, *n*-decane filled microcapsules with different sizes were prepared. The average capsule size of the hydrocarbon-filled microcapsules can be adjusted by modifying the stirring speed during the dispersion process, higher stirring speeds lead to a decrease in capsule size [14].
It could be observed that the combustion rate decreases steadily with an increasing capsule size (Figure 6). It is likely that this behavior is related to the more homogeneous distribution of smaller microcapsules in the hydrogen peroxide gel. Consequently, there is a narrower distribution of local minima and maxima and the fuel can burn optimally without being too thick or too lean locally. Also, smaller capsules have a higher total surface area which can generally lead to higher reaction rates.

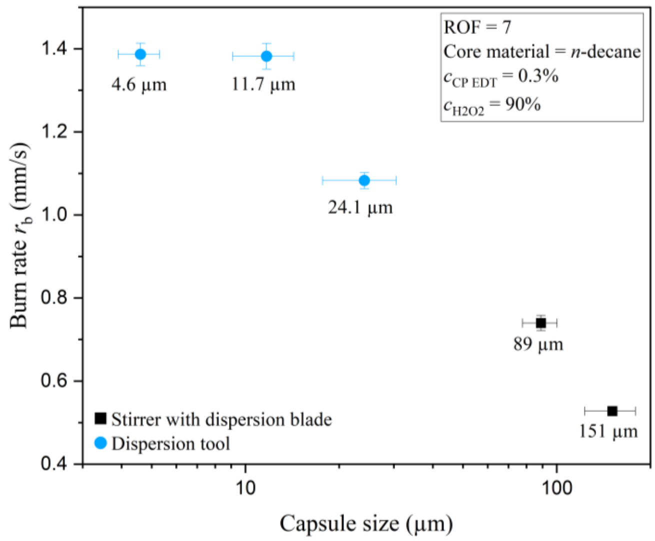

Figure 6: Average combustion rate in dependence of the capsule size (hydrogen peroxide concentration: 90%, ROF: 7).

3.3 Variation of the hydrogen peroxide concentration

In order to evaluate the influence of the concentration of hydrogen peroxide, combustion tests were conducted with five different concentrations (80, 85, 90, 95 and 98%). The results obtained can be seen in Figure 7. As it would be expected, the burn rate increases with increasing hydrogen peroxide concentration and reaches its highest at 98%.

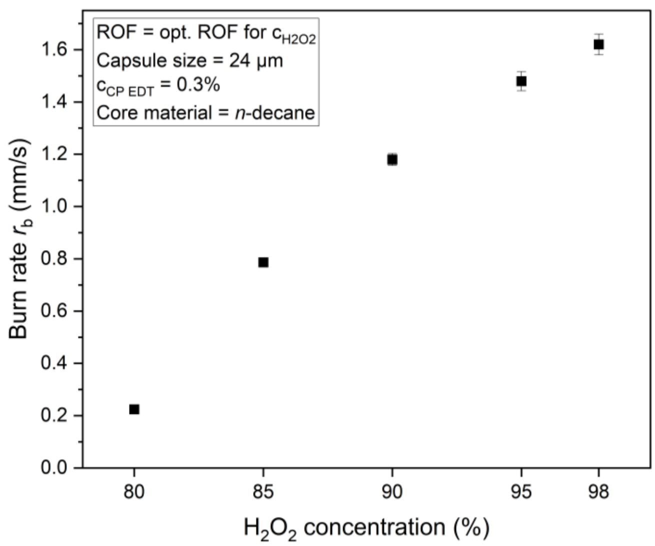

Figure 7: Burn rate in dependence of the hydrogen peroxide concentration.

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3.4 Pressure dependency

The combustion behavior of the microcapsule/hydrogen peroxide monopropellant was also investigated at elevated pressures, to simulate the conditions inside a combustion chamber. The relationship between the burn rate (r_b) and the pressure (P) is commonly described by Vielle's law (also known as Saint Robert's Law, Equation 1).

$$\boldsymbol{r_b = a\, P^n} \tag{1}$$

In Vielle's law, a is a preexponential burn rate coeficient related to properties of the propellant and n is the pressure exponent, inidicating the sensitivity of the burning rate to changes in pressure. High values of n can indicate that the propellant can undergo a self-accelerating process, in which small changes in chamber pressure lead to a significant increase in burn rate, which inevitably increases chamber pressure, leading to instabilitys in the combustion or in the worst case to a disastrous outcome [17,19]. The experimental data and regression analysis is shown in Figure 8.

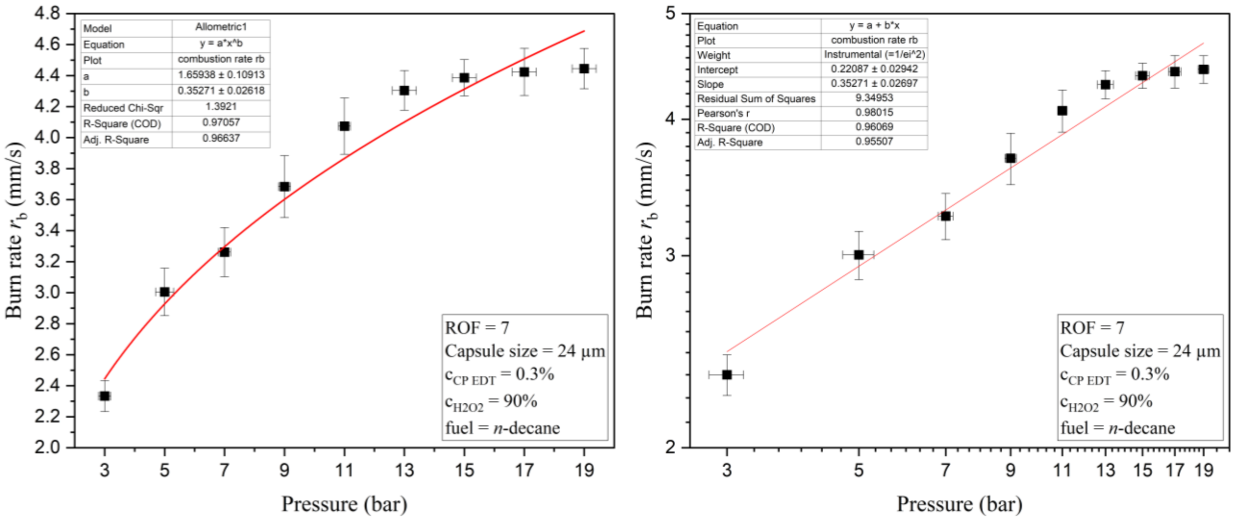

Figure 8: Average burn rate of n-decane filled microcapsules in dependence of the pressure (left) and the corresponding logarithmic plot (right).

The pressure exponent was determined with $n = 0.35$, which is in the range of typical solid propellants [20]. The burn rate coeficient was found to be at $a = 1.66 \frac{mm}{s\,bar^n}$. In the logarithmic plot of the pressure dependency data (Figure 8, right), the values from 3 to 13 bar show a strong linear trend, but for the values above 15 bar, an allusion of the curve plateauing can be seen. This pattern of pressure-independent behavior is common among many propellants [20]. However, more data is needed at pressures greater than 20 bar to confirm this. Based on this data, a first assessment of combustion characteristics for future hot-fire tests can be conducted.

4. Conclusion and Outlook

In this work, a critical step in the development of a new high performance monopropellant consisting of fuel-filled microcapsules dispersed in hydrogen peroxide was taken by conducting a first series of combustion experiments in a strand burner setup to evaluate the influence of different propellant parameters on the burn rate. First, we were able to show, that the ROF of the highest experimental burn rate is indeed associated with the ROF at the maximum calculated specific impulse. With regard to the hydrogen peroxide concentration used, a decline in the burn rate was observed with decreasing hydrogen peroxide concentration. An increase of the burn rate could be achieved by a decrease of the average capsule size. In addition combustion experiments under increased pressure were conducted and showed, that higher pressures led to an increase of the burning rate. These results are of particular interest for the further development of the propellant and for later hot-fire tests. In further experiments, we intend to investigate additional parameters, such as the use of different fuels inside the microcapsules and to evaluate other ignition methods such as catalysts.

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CRediT authorship contribution statement

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

Felicitas Moll: Investigation, Writing – original draft.

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

Stefan Schlechtriem: Supervision.

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