# Hybrid Mixture Explosions Testing in the 1 qm Vessel

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# 1 Hybrid Mixture Explosions - the Nex-Hys Project

The Nex-Hyx Project started in 2019 an intended to create standardizable determination methods for safety-related parameters of explosion protection for hybrid mixtures. Ending in 2022, the work on the project is ongoing in a German standardization committee (NA 095-02-09-01 AK). Reporting about the starting phase a work in progress poster was presented at the ICDERS in Peking 2019. The current paper is summarizing the internationally yet unpublished findings in the 1 qm vessel that were done by the project partner INBUREX Consulting GmbH.

Whereas the current standards for explosion protection usually treat the phases Gas and Dust separately both do occur at the same time in industry. As it is long [1] known that such hybrid mixtures behave differently the project addressed the necessity to develop methods to measure:

- max. explosions pressure
- max. pressure rise velocity
- LEL/MEC
- LOC
- MIE
- MIT

Form the very beginning it became clear that a new method for the minimum ignition energy (MIE) will not be be developed within the project and will have to wait for future activities [2]. The research on the minimum ignition temperature (MIT) was separated, as the experimental setup (the Godbert-Greenwald oven) is different from the explosion vessels used for the remaining quantities (results see [3]). The 20 liter sphere became to preferred tool in contrast to the dust explosion standard were the 1 qm vessel is still the basis. Extensive mesurements in the 20 liter sphere were done and published in the last years [4], [5].

The data summarized here is the result of the experimental work done by the INBUREX Consulting GmbH throughout the project and an important point of orientation as they are the only project partner that could perform measurements in the 1 qm vessel. Data for comparison within the project and in published international literature [6] is widely missing. Results for two hybrid mixtures with cornstarch as dust are presented. One is with a gas (Methane) and the second with a vapor (Isopropanol).

## 2 1 qm hybrid explosion testing setup

The explosion tests were carried out on the  $1 \text{ m}^3$  vessel. The test apparatus is set up on the test site of INBUREX Consulting GmbH. By installing it under a cantilever roof, there is protection against extreme weather influences such as wind, rain and snow. Nonetheless, the experiment is exposed to the environmental temperature, thus experiments in winter were not possible.

The  $1 \text{ m}^3$  vessel is designed and equipped according to the specifications of DIN EN 14034 and consists of the components show in Fig. 1.



Figure 1: 1  $m^3$  vessel at the INBUREX test site.

Labels in Fig. 1:

- 1. dust storage bin
- 2. Ignition lance with pyrotechnic detonators
- 3. Ring nozzle for dust introduction/distribution
- 4. Piezoelectric pressure sensors
- 5. relief valve

The three piezoelectric pressure transducers record the pressure, the pressure rise over time and the absolute pressure. The time resolution is 0.5 ms. The ignition delay time, the time between the introduction of the dust and the ignition of the pyrotechnic ignitors is 600 ms. After the tests, the three measurement curves were checked for plausibility and deviations from one another. The pressure increase over time is determined from the time derivative of the pressure curves - the median value between two of the three maximum values results in the maximum pressure increase value.

To generated the hybrid mixtures the fuel gas (Methane) is metered into the vessel via a mass flow controller.

To generate the Isopropanol vapor, the liquid was placed in a pressure vessel. The container was then subjected to an excess nitrogen pressure of 15 bar and heated to a liquid temperature of 150°C. The liquid was then fed into the 1 m<sup>3</sup> vessel via another mass flow controller. In the injection area, the Isopropanol evaporates immediately.

Methane and Isopropanol were standard grade chemicals. The starch was procured for all project partners in the beginning of the project and is characterized by the data in Tab 1.

1 qm hybrid explosions

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Value	Corn Starch	
humidity	6.8 % by weight	
particle size distribution	D10: 2µm // D50: 13µm // D90: 19µm	
	$20\%$ of the sample is smaller than 10 $\mu\mathrm{m}$	
minimum ignition energy (without inductivity)	100  mJ < MZE < 300  mJ	
minimum ignition energy (without inductivity)	30  mJ < MZE < 100  mJ	
lower explosion limit	$40 \text{ g/m}^3$	
max. explosions pressure (10 kJ// 2 kJ)	8.4 bar // 7.7 bar	
max. pressure rise velocity	580  bar/s	

Table 1: Characteristics of the Nex-Hys corn starch.

# 3 Hybrid explosion heat maps

The following figures show a summary of the test results with an ignition energy of 10 kJ and 2 kJ (pyrotechnic igniters) for corn starch / methane. Each concentration was repeatedly determined according to the test plan on different measurement days in order to determine the reproducibility of the measurements.

The explosion pressure in the tests depends on the pressure at ignition and the effectiveness of the material conversion. In the hybrid mixture, an additional fuel is present in the test volume, which also consumes oxidizer (here atmospheric oxygen) during the reaction. The dosage selected increases the inlet pressure in the test tank by up to 100 mbar. It is not standard practice to evacuate large containers such as 1m<sup>3</sup> before ignition. This additional pre-pressure increases the maximum explosion pressure by up to 1 bar - the pressure reached is therefore 10% above the expected value for the respective concentration. The trend in the explosion pressures is comparable to the rates of pressure rise, but generally shows smaller variation over the concentration ranges. However, this does not affect the areas of the lower explosion limits.

For the maximum explosion overpressure, it was examined whether, a smaller ignition energy of 2 kJ has an impact on the value and position of the optimal mixture. Fig. 2 shows the results of these tests.



Figure 2: hybrid maximum explosions pressure in the 1  $m^3$  vessel for 2 kJ (left) and 10 kJ (right) ignition energy.

The ignition energy has little influence on the achievable explosion pressure, the values only

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differ at low concentrations.

Fig. 3 shows the pressure rise velocity for the hybrid mixture system. The result is that the highest values for the rate of pressure rise are below the optimum concentrations of the pure substances. The maximum value of the rate of pressure rise was reached at a dust concentration of  $125 \text{ g/m}^3$  and a gas concentration of  $90 \text{ l/m}^3$ . The magnitude of the values for only 2kJ ignition energy are comparable, but on average 10% below the measurements with 10 kJ.



Figure 3: hybrid maximum pressure rise velocity in the 1 m<sup>3</sup> vessel for 2 kJ (left) and 10 kJ (right) ignition energy.

The greatest change could be experienced in measurements at the edge of the lower explosion limits, where the ignition probability fell below 100% and up to four repetitions were required for a measurable pressure increase.

Tab. 2 summarizes the test results for Starch / Isopropanol mixtures with an ignition energy of 10 kJ. Here no test with 2 kJ were performed.

Table 2: hybrid explosion characteristics of Starch / Isopropanol mixtures: explosions pressure [bar] / pressure rise velocity [bar m s-1].

Isopropanol [g]			Cornstarch [g]		
	0	250	500	750	1000
11			9,32 / 230		
55		8,07 / 165	9,30 / 278	8,69 / 259	
88	6,90 / 154		9,58 / 319	9,20 / 295	8,10 / 239
121	5,87 / 41	9,46 / 296		8,64 / 330	

The result is that the highest values for the rate of pressure rise are above the optimum concentrations of the pure substances (257 bar\*m/s at 500 g/m<sup>3</sup>). The maximum value of the rate of pressure rise was reached at a dust concentration of 750 g/m<sup>3</sup> and a gas concentration of 121 g/m<sup>3</sup> and is well below the values reached for hybrid dust/gas mixtures.

## 4 Conclusion

As part of the Nex-Hys project, around 240 tests were carried out in the  $1 \text{ m}^3$  vessel and further tests to verify the results in the 20 l sphere. The following conclusions could be drawn from the experiments:

- It could be shown that the maximum values (pressure / rate of pressure increase) for the hybrid system corn starch / methane are reached at concentrations below the optimal concentrations for the pure substances. This was expected because there is in total more fuel in the hybrid mixture. The optimal combustion point is shifted to lower concentrations.
- In the hybrid mixture, the maximum pressure rise velocity is mainly determined by the most reactive pure component (in the turbulent state).
- The maximum explosion pressure in the 1 m<sup>3</sup> vessel is largely independent of the ignition energy (2 kJ / 10 kJ). The deviation is around 10%.
- The pressure in the container before ignition has a major influence on the maximum explosion pressure and the pressure rise velocity. For larger experimental facilities (>20 l), the increase in pressure due to the introduction of fuel has not been taken into account so far.
- For containers up to  $1 \text{ m}^3$ , the mixing of the gas phase by the introduction of dust is sufficient no segregation takes place until ignition.

Additionally, it must be stated that hybrid mixtures with vapors of liquids that are condensable at room temperature can only be measured to a limited extent in large vessels. Vapor concentrations (2 to 15% by volume) for liquids can only be tested if the test containers can be heated directly and the temperature field in the container is homogeneous. For large containers without heating, the dosing time is greater than the duration for the condensation of liquids from the gas phase. This can be seem by the shift of the maximum values in Tab. 2 to higher concentrations that indicate the condensation of the vapor.

As result of the Nex-Hys project not only the standardization process (in Germany) is initiated [7] but in parallel international round robin tests are carried out to validate the proposed procedures and findings. The first round robin test for hybrid gas/dust mixtures is already completed [5] and the second has stared, as well as the first round robin test for hybrid vapor/dust mixtures.

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