CHEMICAL INHIBITION OF PREMIXED HYDROGEN-AIR FLAMES: EXPERIMENTAL INVESTIGATION USING A 20-LITRE VESSEL

van Wingerden, M.[1], Skjold, T.[1], Roosendans, D.[2], Dutertre, A.[2] and Pekalski, A.[3]
[1] Department of Physics and Technology, University of Bergen, Allégaten 55,
PO Box 7803, 5020 Bergen, Norway, mattheus.van.wingerden@uib.no
[2] Total, 24 Cours Michelet, Tour Michelet, 92069 Paris La Défense Cedex, France
[3] Shell International Petroleum Company Limited, Shell Centre, London SE1 7NA,
United Kingdom

ABSTRACT

Throughout the history of the mining, petroleum, process and nuclear industries, continuous efforts have been made to develop and improve measures to prevent and mitigate accidental explosions. Over the coming decades, energy systems are expected to undergo a transition towards sustainable use of conventional hydrocarbons and an increasing share of renewable energy sources in the global energy mix. The variable and intermittent supply of energy from solar and wind points to energy systems based on hydrogen or hydrogen-based fuels as the primary energy carriers. However, the safety-related properties of hydrogen imply that it is not straightforward to achieve and document the same level of safety for hydrogen systems, compared to similar systems based on established fuels such as petrol, diesel and natural gas. Compared to the conventional fuels, hydrogen-air mixtures have lower ignition energy, higher combustion reactivity, and a propensity to undergo deflagration-to-detonation-transition (DDT) under certain conditions. To achieve an acceptable level of safety, it is essential to develop effective measures for mitigating the consequences of hydrogen explosions in systems with certain degree of congestion and confinement. Extensive research over the last decade have demonstrated that chemical inhibition, or partial suppression, can be used for mitigating the consequences of vapour cloud explosions (VCEs) in congested process plants. Total and cooperation partners have demonstrated that solid flame inhibitors injected into flammable hydrocarbon-air clouds represent an effective means of mitigating the consequences of VCEs involving hydrocarbons. For hydrogen-air explosions these same chemicals inhibitors have not proved effective. It is however well-known that hydrocarbons can affect the burning velocity of hydrogen-air mixtures greatly. This paper gives an overview over previous work on chemical inhibitors. In addition, experiments in a 20-litre vessel have been performed to investigate the effect of combinations of hydrocarbons and alkali salts on hydrogen/air mixtures.

Introduction

Over the coming decades, energy systems are expected to undergo a transition towards an increasing share of renewable energy sources in the global energy mix. Hydrogen or hydrogen-based fuels are expected to become the primary energy carriers. However, the safety-related properties of hydrogen imply that it is not straightforward to achieve and document the same level of safety for hydrogen systems, compared to similar systems based on established fuels such as petrol, diesel and natural gas. Compared to the established fuels, hydrogen has considerably lower ignition energy, higher combustion reactivity, a wider range of flammable fuel-air concentrations, and a propensity to undergo deflagration-to-detonation-transition (DDT). To achieve an acceptable level of safety, it is essential to develop effective measures for mitigating the consequences of hydrogen explosions in systems with certain degree of congestion and confinement. Extensive research over the last decade have demonstrated that chemical inhibition, or partial suppression, can be used for mitigating the consequences of vapour cloud explosions (VCEs) in congested process plants. Total and cooperation partners have demonstrated that solid flame inhibitors injected into flammable hydrocarbon-air clouds represent an effective means of mitigating the consequences of VCEs involving hydrocarbons.

This paper reviews previous work on chemical inhibition of fuel-air mixtures, with particular emphasis on hydrogen, and presents results obtained from experiments performed to investigate the effect of solid flame inhibitors in combination with a (in fact) flammable compound on the maximum explosion pressure, maximum rate of pressure rise, and laminar burning velocity, for premixed combustion of

hydrogen-air mixtures. The results were obtained in a 20-litre vessel at the standard initial conditions of turbulent flow normally used for determining the properties of flammable dust-air mixtures.

Previous work

An inhibitor is a substance that is added to a fuel-air mixture to reduce the burning velocity of the mixture. The burning velocity can be reduced by inerting, cooling or chemical inhibition. Inerting is adding a gas that does not chemically react with the fuel or air. The gas will displace the air and hence limit the amount of oxygen in the mixture. Common inerting gases used in the industry are nitrogen, carbon dioxide and argon.

The overpressure in chemical explosions is generated by the rapid expansion of post combustion gases due to the heat generated in exothermal reactions. Reducing the combustion temperature will lower the explosion overpressure. This can be achieved by adding substance with a higher heat capacity than the air/fuel mixture. For example, carbon dioxide is a more effective inert gas compared to nitrogen due to the higher heat capacity. Adding a liquid or solid which will undergo a phase transition and/or decompose is another way of reducing the temperature increase. An example is the use of water deluge at offshore platforms to limit the consequences of gas explosions [1].

Chemical inhibition are substances that interfere chemically with the combustion process, which is mainly driven by chain branching reactions. The combustion of hydrogen in air involves many elementary reactions that generate radicals such as H· and OH·, which are combustion enhancing radicals. The introduction of a substance that reacts with these radicals can substantially reduce the burning velocity of the premixed mixture. Chemical inhibitors can be very effective, even with relatively small amounts added.

Over the last 100 years, extensive research has been performed on chemical inhibitors. Babushok *et al.* [2] summarised research for alkane combustion where different types of inhibitors were ranked based on experiments and simulations. The most effective inhibitor for alkane combustion is iron pentacarbonyl ($Fe(CO)_5$), but sodium- and potassium bicarbonate are also very effective. Other suppressants, including halons (halon 1011) and mono-ammonium phosphate, were shown to be effective at a smaller scale for fuels with a K_G-value[1] of 200 bar·m/s and 400 bar·m/s, respectively [3]. Mono-ammonium phosphate was shown to be effective for propane ($K_G \leq 100$ bar·m/s) for volumes up to 250 m^3 [4]. Explosion suppression is a technology where an explosion is detected during its early stages (most commonly by monitoring pressure) and extinguished by injecting a sufficient quantity of flame inhibitor (suppressant) (pressurised nitrogen is typically used as propellant) in a short time interval.

Hoorelbeke [5] developed technology for mitigating vapour cloud explosions by injecting flame inhibitors into the flammable vapour cloud before ignition. The technology implies application of a gas detection system to recognise a dangerous vapour cloud developing and an appropriate inhibitor injection system [6]. He showed that potassium carbonate can be used as a flame inhibitor for various hydrocarbons: methane, ethane, propane, butane, ethylene, propylene, butylene and even acetylene. He also demonstrated the effectiveness of potassium bicarbonate and sodium chloride as flame inhibitors for propane, and confirmed the effectiveness of sodium bicarbonate as an inhibitor for propane. However, the same inhibitors had hardly any effect on the rate of combustion for hydrogen-air mixtures.

The combustion of hydrogen is inherently different from that of hydrocarbons. Inhibitors found to be effective for alkanes only have a limited effect on hydrogen. For example, iron pentacarbonyl only gives a slight reduction of the flame velocity of hydrogen [7]. Hoorelbeke [5] observed the same for potassium carbonate.

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[1] The K_G-value is maximum rate of pressure rise normalized to a volume of 1 m^3 using the so-called cube-root-law: $K_G=(dp/dt)_{max}.V^{1/3}$ = constant with $(dp/dt)_{max}$ = the maximum rate of pressure rise in volume V.

Miller *et al.* [8] studied the effect of various gaseous and liquid inhibitors on premixed combustion of hydrogen-air mixtures. The inhibitors tested were classified into 3 groups:

Good: saturated and unsaturated hydrocarbons, mono halogenated hydrocarbons, metal carbonyls, some metal chlorides

Fair: hydrogen halides, inorganic sulphur compounds, partially fluorinated and chlorinated hydrocarbons, some metal halides

Poor: halogens, fully fluorinated and chlorinated hydrocarbons, oxides of carbon and nitrogen, silicon and boron halides

Some of the most effective inhibitors found during the study by Miller *et al.* [8] were saturated and unsaturated hydrocarbons. This was confirmed in an experimental study performed by Schröder *et al.* [9] who performed experiments in a closed 14-litre vessel for mixtures of methane and hydrogen. The results, presented as the maximum rates of pressure rise normalized to a volume of 1 m³ (K_G-value) show that the reactivity of methane-hydrogen mixtures is similar to that of pure methane when the methane content of the mixture is 50 % or higher. This result was confirmed by Ilbas *et al.* [10] who measured the laminar burning velocity of methane-hydrogen mixtures: a relatively moderate increase of laminar burning velocity was seen when increasing from pure methane to a 50% methane-50 % hydrogen mixture whereas the laminar burning velocity increased much more beyond this mixture composition (see Figure 1).
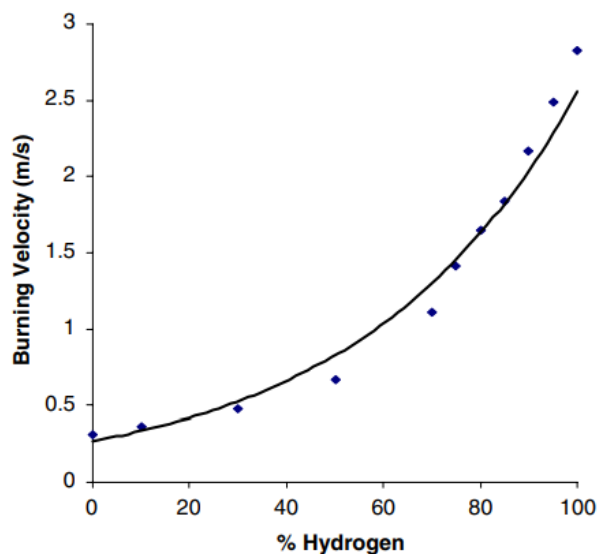

Figure 1   Burning velocity of stoichiometric methane-hydrogen mixtures with air (from Ilbas et al., 2006 [10])

Large scale experiments with methane-hydrogen-air mixtures have been performed during the NATURALHY project [11]. These experiments were performed in a 3 x 3 x 2 m framework stacked with grids of metal bars. Tests were performed with 0-100, 25-75, 50-50, 75-25 and 100-0 mixtures of hydrogen-methane in air. The overpressure generated in these experiments indicated similar results as those found in the lab-scale experiments.

Jithin *et al.* [12] confirmed the findings of Miller *et al.* [8] for butane-hydrogen mixtures with up to 60 % hydrogen.

For both hydrocarbon and hydrogen combustion one of the most sensitive reactions is [13]:

$$H + O_2 \rightarrow O + OH$$
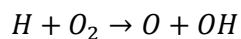
This reaction is chain branching and are often followed up by reaction that regenerate H radicals and make the reaction explosive. In hydrogen flames the generation of H radicals is 10 times higher than for methane, see Figure 2. Even though inhibitors like alkali salts influence the same flame radicals for both hydrocarbon and hydrogen combustion, the latter requires significantly more to be effective [14].
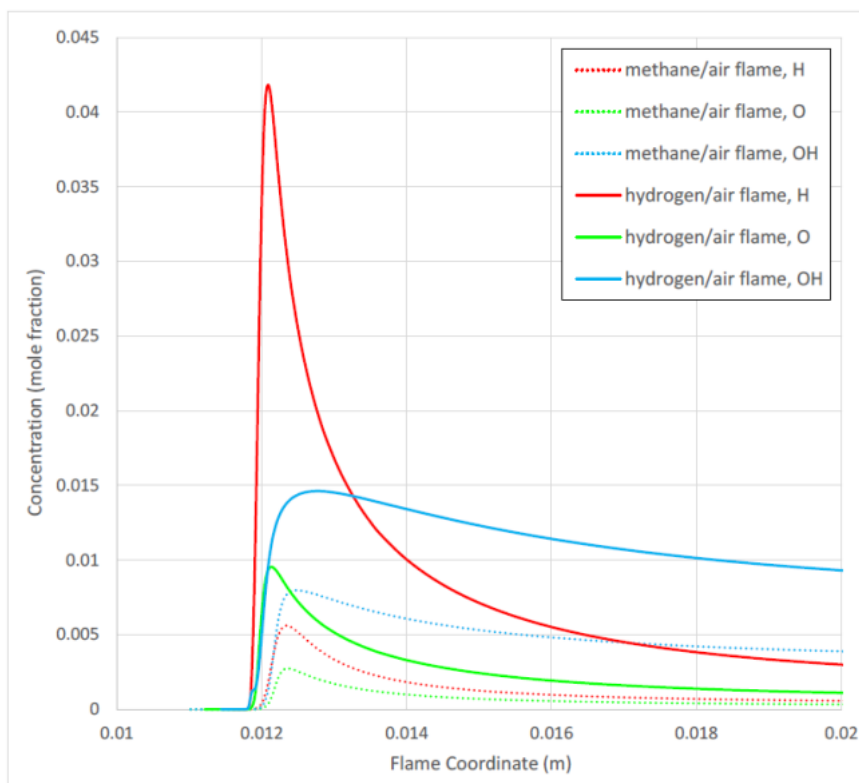

Figure 2   Predicted overshoot of flame radicals in stoichiometric methane/air and hydrogen/air flames (from Roosendans [14])

Addition of alkanes into hydrogen/air mixtures yields reactions between H radicals and hydrocarbons. The reaction rates of these are often larger than for the branching reaction for H radicals (1). As these effectively compete with the branching reaction for H radicals, the chain branching rate will be reduced [13]. This makes hydrocarbons work as inhibitors.

In the light of these results an experimental study was performed using propane as the main flame inhibitor. In addition, sodium bicarbonate was used to investigate if the effect can be enhanced using working inhibitors for hydrocarbons.

Experimental set-up

The experiments were performed in a 20-litre closed vessel. The vessel is equipped with a 0.6-litre dispersion chamber. Prior to ignition, this dispersion chamber is filled with 20 barg air and released into the 20-litre vessel. The time between onset of dispersion and ignition was 60 ms. For the experiments to be performed under atmospheric conditions, the 20-litre vessel was evacuated to 400 mbar absolute pressure. Gases were added to the 20-litre vessel using partial pressure, and solids were added to the dispersion chamber. This gave the set-up some limitations with respect to concentrations of the added gases. Since the minimum pressure obtainable with the vacuum pump was 90 mbar absolute pressure,

the sum of added gases could not exceed 31 volume percent. To have room for added propane, the tests were performed with 28% hydrogen, thus a slightly lean mixture with respect to hydrogen (the propane was added in addition).

The effective particle size of the solid inhibitor is crucial [15]. The particles should be as small as possible. Models indicate that particles sizes of 3µm and 16 um for NaHCO₃ would be required for full inhibition of hydrogen and methane, respectively [16]. Larger particles may still work, but the effectiveness is greatly reduced as the particles size increases. Figure 3 shows the particles size distribution for the inhibitors used in this work. For the sodium bicarbonate, roughly 20% of the inhibitor is below 10µm. Hence, the dust is too coarse to have a real impact on the hydrogen/air mixtures, but it should be suitable for hydrocarbon/air mixtures. The potassium carbonate used was a much finer powder. With more than 95% being below 10µm, this inhibitor should be well suited for hydrogen/air mixtures.
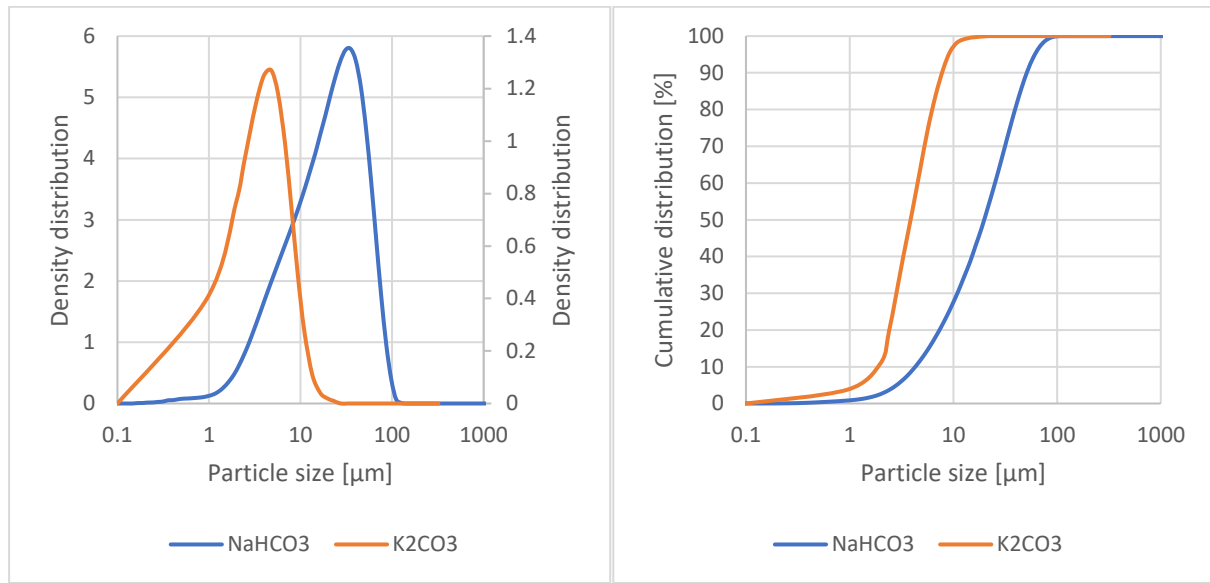

Figure 3 Particles size distribution of sodium bicarbonate and potassium carbonate used in the experiments.

Most mixtures were ignited by a spark generator. The energy of the discharge is estimated to approximately six joules [17]. A 1kJ chemical ignitor was used for the mixture that could not be ignited by the 6 J discharge. The mixtures in mention were those with 2 and 3% added propane and inhibitor loading of 150g/m³. Additionally, for potassium carbonate, the tests with the same amount of added propane and 50g/m3 inhibitor loading had to be ignited with a chemical ignitor. The pressure was monitored using two piezoelectric sensors and a sampling rate of 2500 Hz.

Calculations of the laminar burning velocity

The 20-l vessel used in this work is a standardized set-up often used for determination of the properties of flammable dust-air mixtures. The main advantage of the set-up being standardized is that models have been developed and the turbulence levels in the vessel have been measured. This makes it possible to estimate the laminar burning velocity using values extracted from the pressure-time curves.

The burning velocity of the turbulent flame ($S_T(t_{ip})$) at the inflection point can be calculated using a thin flame approximation [18]:

$$S_T(t_{ip}) = \frac{1}{3(p_f-p_i)}\left(\frac{dp}{dt}\right)_m V_v^{1/3} \left(\frac{3}{4\pi}\right)^{1/3} \left(\frac{p(t_{ip})}{p_i}\right)^{-1/\gamma} \left\{1 - \left(\frac{p_f-p(t_{ip})}{p_f-p_i}\right)\left(\frac{p(t_{ip})}{p_i}\right)^{-1/\gamma}\right\}^{-2/3} \quad (2)$$

Where $p(t_{ip})$ is the pressure at the inflection point, p_i is the initial absolute pressure, p_f is the final absolute pressure, γ is the specific heats ratio (assumed to be equal to 1.40) and V_v is the volume of the explosion vessel (20 l).

The correlation between the laminar burning velocity S_L and the turbulent burning velocity can be written as [19]:

$$S_T = 15.1 \cdot S_L{}^{0.748} \ u'_{rms}{}^{0.412} \ \ell_I{}^{0.196} \quad (3)$$

Where u'_{rms} is the turbulence intensity and ℓ_I is the integral turbulence length scale.

An estimate for the turbulence intensity at the inflection point was derived by Dahoe [20]:

$$u'_{rms}(t_{ip}) = u'_{rms}(t_0) \left(\frac{t_{ip}}{t_0}\right)^{-1.61} \quad (4)$$

Where $u'_{rms}(t_0)$ is the turbulence intensity at the time of ignition, found to be 3.75 m s^{-1}, t_{ip} is the moment of occurrence of the inflection point and t_0 the moment of ignition relative to the moment of the start of introducing the inhibitor (in this case t_0=0.060s).

The corresponding estimate for the turbulent integral length scale is [21]:

$$\ell_I(t_{ip}) = \ell_I(t_0) \, exp\left(a_1 \, ln\left(\frac{t_{ip}}{t_0}\right) + a_2 \left\{ln\left(\frac{t_{ip}}{t_0}\right)\right\}^2\right) \quad (5)$$

Where $a_1 = -3.542$, $a_2 = 1.321$, $\ell_I(t_0) = 0.012845$ m and $t_0 = 0.0588$ s.

An empirical relation corrects the measured overpressure (p_{ex}) for cooling effects to the vessel walls and the influence of pyrotechnic igniters [22]:

$$p_m = \begin{cases} 5.5(p_{ex} - p_{ci})/(5.5 - p_{ci}) & \text{when} \quad p_{ex} < 5.5 \text{ bar} \\ 0.775 p_{ex}{}^{1.15} & \text{when} \quad p_{ex} > 5.5 \text{ bar} \end{cases} \quad (1)$$

Where p_{ci} is the overpressure caused by the chemical igniter.

A measure of laminar burning velocity follows from an inverse version of Eq. 3, using the estimated values obtained from Eqs. 2, 4 and 5:

$$S_L(t_{ip}) = 0.0315 \cdot \left[S_T(t_{ip})\right]^{1.276} \cdot \left[u'_{rms}(t_{ip})\right]^{-0.526} \cdot \left[\ell_I(t_{ip})\right]^{-0.250} \quad (2)$$

According to Dahoe [23] the described method results in estimates of laminar burning velocities which are approximately 5-10 % higher than established values of laminar burning velocities of hydrocarbon fuels.

Results and discussion

Figure 4 shows that the addition of propane and sodium bicarbonate only had a slight impact on the maximum overpressure generated inside the vessel. Adding small quantities of propane slightly adds to the overpressure. Additional propane addition decreases the maximum overpressure. Addition of sodium bicarbonate also slightly reduced the overpressure. The overpressure is mainly depended on the stoichiometry of fuel and oxidiser in addition to the cooling of the system. Adding propane to the system adds more fuel and increases the overpressure until oxygen is the limiting factor of the system. Addition of 2 and 3% propane decreases the amount of oxygen in the system resulting in a slightly lower overpressure. Reduction of the overpressure when adding sodium bicarbonate or potassium carbonate

is most likely due to cooling of the system. Both sodium bicarbonate and potassium carbonate undergo several phase transitions and decompositions requiring heat.

Figure 5 and Figure 6 show the maximum rate of pressure rise and the laminar burning velocity, respectively. The calculations of the burning velocity depend on both the maximum overpressure and maximum rate of pressure rise. As the inhibitors only have a slight effect on the maximum overpressure, the laminar burning velocity follows the same trend as the maximum rate of pressure rise.
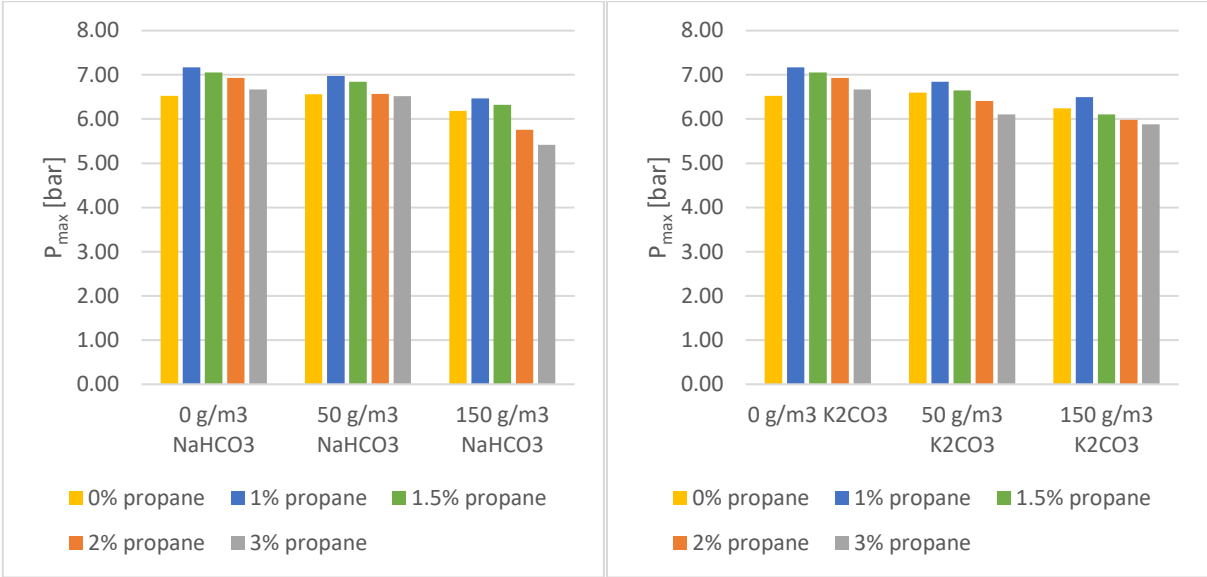

Figure 4   Effect of propane and solid inhibitor on the maximum overpressure for hydrogen-air mixtures.

The laminar burning velocity calculated for 28% hydrogen in air is around 170 cm/s. This is slightly lower than the expected value of around 200 cm/s [24]. This is possibly due to the model used for calculating the burning velocity and equipment used in these experiments being slightly different from the equipment used to make these models. The laminar burning velocity is still presented for easier comparison with other experiments.
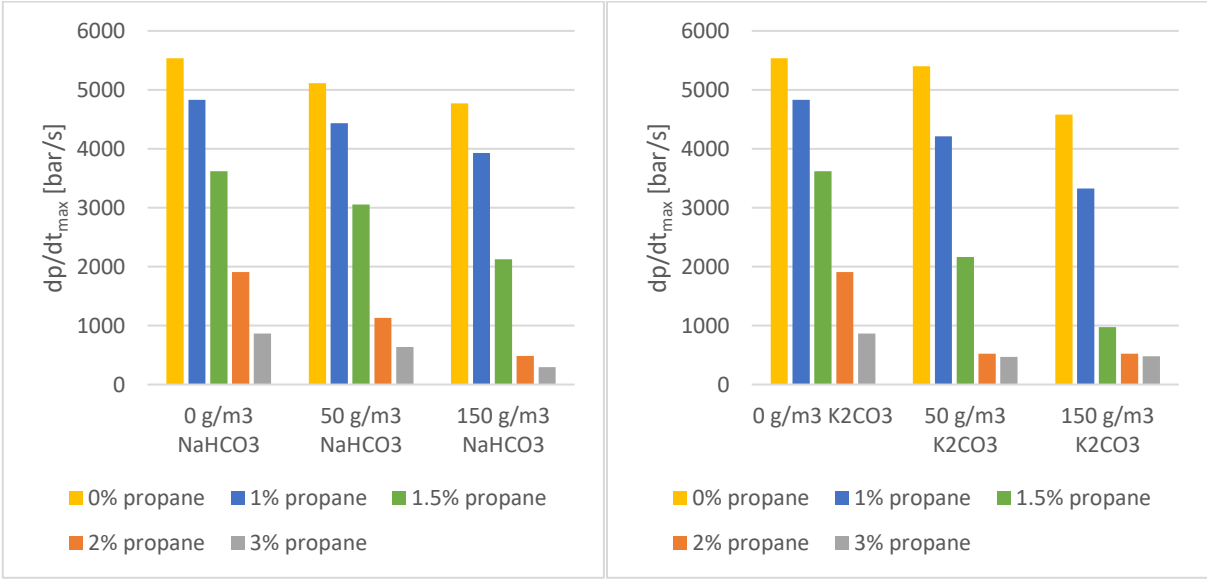

Figure 5   Effect of propane and solid inhibitor on the maximum rate of pressure rise for hydrogen-air mixtures.

Addition of propane greatly reduces the laminar burning velocity of the mixture. For addition of 2 and 3% vol propane a reduction of the laminar burning velocity of 70% and 85% respectively is observed. This is in line with observations from previous work. In the case of 2% propane, the laminar burning velocity was reduced to 50 cm/s, which is close to the laminar burning velocity of pure propane.

Both sodium bicarbonate and potassium carbonate reduced the laminar burning velocity slightly. Addition of 150 g/m3 yielded a reduction of around 8% and 11% for sodium bicarbonate and potassium carbonate, respectively.
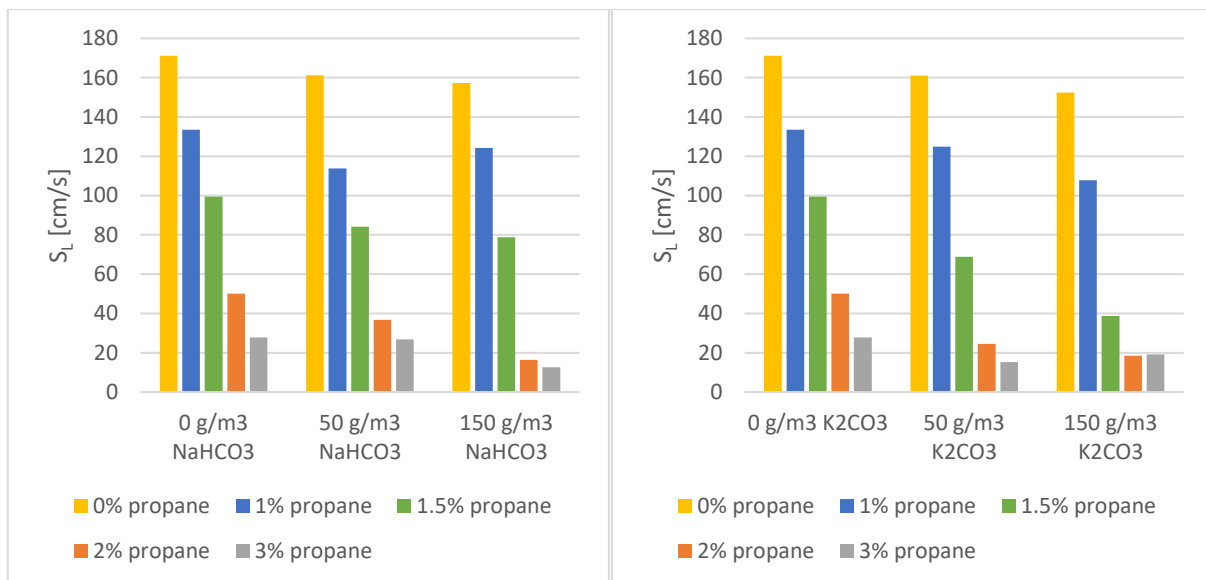

Figure 6   Effect of propane and solid inhibitor on the laminar burning velocity for hydrogen-air mixtures.

The focus was the effect of sodium bicarbonate and potassium bicarbonate on the already reduced laminar burning velocity of the hydrogen-propane-air mixtures. For the test with addition of 3% propane, the laminar burning velocity was reduced from 28 cm/s to 13 cm/s with addition of sodium bicarbonate. It is worth noting that these 2 tests were ignited by different ignition sources, the ignition source for the latter one was almost 200 times stronger. As stronger ignition sources often yield stronger explosions, the inhibition effect might even be stronger. The same was observed with potassium carbonate. It however only required 50 g/m^3 to achieve full inhibition with potassium carbonate apposed to the 150 g/m^3 needed for sodium bicarbonate. This is most likely due to the large number of fine particles in the potassium carbonate powder used.

For the case with 2% propane, the effect of sodium bicarbonate was stronger than for the case with 3% added propane, reducing the laminar burning velocity from 50 cm/s to 16 cm/s which is a 70% reduction. Again, the same was observed for potassium carbonate.

For 1% and 1.5% propane no significant effect was observed for sodium bicarbonate. Potassium carbonate yielded a strong reduction for the case with 1.5% added propane, reducing the laminar burning velocity from 100 cm/s to 40 cm/s.

Conclusion

The effects of propane and sodium bicarbonate on hydrogen-air combustion were investigated during this study in the 20L spherical vessel. Adding propane has a significant impact on the laminar burning velocity of the tested mixtures. In line with previous work, relatively small amount can bring the laminar burning velocity close to the burning velocity of the alkane added.

While sodium bicarbonate only has a slight effect on pure hydrogen-air mixtures, it does have a significant effect on hydrogen-air mixtures inhibited by propane. In this work, laminar burning velocities down to 13 cm/s were observed for mixtures with 28% hydrogen, 3% propane and 69% air. The study showed the same effects with potassium carbonate. Potassium carbonate seemed however more effective in mixtures with smaller amounts of added propane.

In this study only a limited number of tests and repetitions were performed. Further studies are required to confirm the findings in this paper. Further studies may include different hydrogen concentrations, a wider range of solid inhibitor, additional types of inhibitor and different turbulence levels.

Acknowledgements

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