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FLAMMABILITY BEHAVIOUR OF POLYDIMETHYLSILOXANE (PDMS) MEMBRANES UNDER NORMOXIC CONDITIONS FOR SPACECRAFT APPLICATIONS

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ABSTRACT

Two types of polydimethylsiloxane PDMS membranes with two thicknesses, 0.125 mm and 0.25 mm, were studied experimentally for a series of environmental conditions in a closed chamber. The conditions were changing oxygen concentration, ambient pressure and normoxic conditions. The results for piloted ignition are linearly dependent on oxygen concentration and ambient pressure via chemistry. A non-monotonic dependency was found in normoxic conditions, but the dominant mechanisms are not clear. The flame spread has a linear dependence on oxygen concentration via chemistry and thermal transfer. Two dependency regimes (linear and asymptotic) emerge for flame spread as a function of ambient pressure. The mechanisms are due to thermal transfer (linear regime) and a combination of thermal transfer and kinetics (asymptotic regime). Flame lengths are exponentially dependent on oxygen concentration via oxygen/fuel supply and soot oxidation. On the contrary, ambient pressure affects flame length non-monotonically via fuel supply (increasing regime) and an excess of oxidiser (decreasing regime). Under normoxic conditions, increasing oxygen concentration had a stronger influence than decreasing pressure with respect to both flame spread and flame length. The extinction conditions at low pressure are due to a combination of increased radiative losses (similar to those of conditions) and kinetic microgravity effects. Furthermore, extinction at low pressures occurred well below normoxic or hypoxic conditions. Thus, the two PDMS materials tested are more flammable under normoxic conditions, which needs to be considered when assessing the fire risk associated with spacecraft design and operation. It remains unclear how these results directly translate to microgravity, where other phenomena might dominate, so future experiments in microgravity are recommended.

1. INTRODUCTION

There is currently a revival of the spacecraft industry and renewed interest in space exploration, both through commercial activities (space tourism) and state-led research activities (extending the ISS lifespan, Chinese space station, Lunar space station, and activities related to exploration of Mars). Such space activities require that fire safety is properly considered in order to safeguard the occupants/users and, perhaps most importantly, the spacecraft infrastructure. In the past, several fire incidents and fire accidents have been reported in orbiting spacecraft (space stations) [1–5]. Previous fire accidents on mock-up spacecraft tests, due to excessive oxidizers, led to a fire safety strategy that relies heavily on reducing the ignition causes (environmental and selection of materials) [3–5]. As such, fire safety strategies on spacecraft can be very stringent compared to those on Earth-based infrastructures.

Current standards are based on the fire response of a particular material exposed to a determined amount of energy; the threshold limit is based on the ignition and subsequent flame spread development (NASA-STD-6001B Test 1 and Test 2) [6]. This standard empirical method is known to have flaws as it is performed under a normal-gravity situation that does not represent microgravity environments found on spacecraft [7]. There are currently ongoing efforts to established improved standard methods based on scientific principles [8].

Additionally, the new human exploration missions to go beyond Low Earth Orbit to the Moon and Mars challenge the current spacecraft design with respect to fire safety. The new designs propose to use normoxic conditions within the habitable spaces, i.e. the oxygen concentration is increased while the ambient pressure is reduced in order to keep the partial pressure of oxygen constant. As such, the effect of normoxic environments on the fire risk of solid materials for spacecraft applications is of significant interest. Currently, on the ISS, the normoxic environment is used during extravehicular activities (EVA). Though it might ease structural spacecraft design, the consequences of a normoxic environment on the flammability behaviour of solid materials are not known. Also, current flammability standard screening tests or future ones do not take into account the normoxic environment.

Previous studies on the effect of normoxic environments have shown somewhat contradictory results. For instance, the forced flow effect on ignition was not relevant under normoxic conditions for a noncharring sample [9] and flame spread results in microgravity have shown to be more flammable in normoxic environments [10]. Thus, there is a programmatic and scientific need to understand and predict the flammability behaviour of materials in normoxic environments.

According to NASA's decadal review, new materials and more complex materials should be studied and investigated [11]. Silicone-based materials are promising for this purpose because they have several properties that are relevant for spacecraft applications [12]. Most importantly, a by-product emerging from thermal decomposition (ignition) and combustion of siloxanes is silica-ash. This silica-ash can deposit on the sample, and thus protect it from thermal exposure [13]. As a result, many silicone-based products and siloxane have been reported to have low flammability under certain environmental conditions [14,15]. Siliconebased materials were tested in the past in microgravity conditions, as attested by Kimzey [16]. More recently, in the Saffire II tests, large samples of a silicone-based material (polydimethylsiloxane or PDMS) resulted in successful ignition, but not sustained flame spread [17,18].

Due to all the above, the current experimental investigation was set up to investigate the flammability behaviour of two PDMS membranes under a range of oxygen concentrations and ambient pressure (including normoxic conditions). The flammability behaviour was covered by including ignition delay time, flame spread rate, flame lengths and near-limit considerations.

2. INTRODUCTION

The material used for the current investigation was the silicone-based membrane denominated polydimethylsiloxane or PDMS. The same material has been tested in the microgravity testing programme Saffire II [17,18]. The dimensions of the sample were 200 x 50 mm, and the thicknesses used were 0.125 mm and 0.25 mm, respectively. The manufacturer is Specialty Silicone Products, Inc., and the product is identified as SSPM823. The manufacturer does not provide the specific thermal properties that are relevant for the thermal studies. Some of the thermo-physical properties were reported by Niehaus et al. [13], such as density (970 kg/m³), specific heat capacity (1050 J/kgK) and ignition temperature (673 K).

The rig employed, the TOPOFLAME (see *Figure 1*), is located at the ZARM facilities in Bremen. The TOPOFLAME is a 1000 L vessel where the atmospheric conditions can be controlled. The ambient pressure can vary from 0 to 0.5 MPa, and the oxygen concentration from 0 to 40%. The diagnostic systems used can also be seen in *Figure 1*. Three cameras were used for visual observations and measurement: A Canon EOS 7000 was placed outside the chamber and its goal was to obtain side-view pictures of the flames. The other two cameras, a VIS camera and an IR camera (8.33f/s), were placed inside the chamber. The ignition system consisted of a 29 AWG Kanthal wire coiled to the top of the sample. The energy released to the different samples was 80 J, equivalent to 8 s of 10 W.



Figure 1 – The left photo shows the assembled TOPOFLAME rig before a test takes place. The right photo shows the internal structure and the diagnostic systems once the rig is opened.

The videos obtained from the various cameras, specifically the VIS frontal camera, were used to quantify the flammability parameters: ignition delay times, flame spread rates, flame length, and extinction limits. The ignition delay times were extracted from the video frames (30 fps). The ignition delay times are defined by the time between the coil energising time and when the first flame was observed. Likewise, from the videos, the flame's leading edge and trailing edges position could be monitored. A Matlab script based on a binary code was then used to quantify the flame position and dimension; thus, the flame spread and flame length could be quantified. Finally, the extinction condition was defined when the flame did not spread more than half of the sample length.

The experimental matrix had two sets of experiments to comply with the objectives of the investigation. 0.125 mm membrane was tested under various ambient pressures and oxygen concentrations to achieve ignition, steady flame spread or extinction. For the latter, the environmental conditions for extinctions were found by reducing either the oxygen or the ambient pressure until extinction was achieved. For normoxic conditions, the 0.125 mm and 0.25 mm membranes were used to evaluate ignition and flame spread behaviour.

3. RESULTS

The experimental results obtained in the TOPOFLAME chamber are presented in the following. In the first section, the ignition delay time results as a function of oxygen concentration, ambient pressure and normoxic conditions are discussed. In the second section, the flame spread behaviour is presented by means of flame spread rates and flame lengths. Finally, the extinction behaviour is discussed by using the near-limit boundary maps.

3.1. Ignition

Figure 2 depicts the ignition delay time results as a function of the oxygen concentration (top panel) and as a function of the ambient pressure (bottom panel). It can be seen that the ignition delay times decrease following a linear dependency on oxygen concentration. Hsieh and Buch [19] obtained similar dependencies on the oxygen concentration for similar silicone elastomers. They suggested that an increased oxygen concentration aids the depolymerisation from the silicones. Such linear dependencies have also been reported for other thermally-thin and thick polymeric fuels for varying oxygen concentrations [9,20–22]. An increase in oxygen increases the gas-phase chemical reactions according to the following expression; $\omega_g = A_g \rho_g Y_{O_2} \rho_f Y_f e^{(-E_g/RT_g)}$ [23]. Or conversely, the chemical time in the gas-phase, $\tau_{ch} = (\rho c_p T)/(E/RT)\Delta h_c A e^{-E/RT}$ [24], is reduced. It is clear that the effect of oxygen on the reaction rates, or chemical time, dominates the ignition delay time for the PDMS as a function of the oxygen concentration.

The ignition delay time also decreases linearly with decreasing ambient pressure, see *Figure 2* (bottom panel). Other studies have reported the same behaviour for thermally-thin cellulosic sheets [9,21] and for thermally-thick samples at low pressures [22]. However, Fereres et al. [25,26] and McAllister et al. [22] reported that the ignition delay time decreased as the ambient pressure was decreased for thermally-thick solid. Their work revealed that the convective heat losses are reduced with decreasing pressure dominating the heat losses (reduced at lower pressures) and transport.

The convective heat transfer coefficient in buoyant condition, $h_c = (GrPr)^{1/4}$, is proportional to the square root of pressure (through the Grashof number). Thus, reducing the pressure should decrease the convective heat losses and increase the heating rate at the fuel surface (being heated by conduction and convection by the coil). However, this effect seems to have little or no influence on the PDMS samples, as seen in *Figure 2*.

The effect of pressure on the chemical and reaction rates happens via gas-phase densities and reduction of mass pyrolysates. Then, chemistry dominates the ignition delay times for pressure as a variable, see *Figure 2* (bottom panel).



Figure 2 – Ignition delay time for a 0.125 mm PDMS sample as a function of two environmental parameters, the oxygen concentration (top panel) and ambient pressure (bottom panel).

The effect of the normoxic environment on the ignition delay time is depicted in Figure 3. For the 0.125 mm sample, the ignition delay time exhibits a nonmonotonic behaviour, and ignition occurs more readily at 0.7 bar and 30% oxygen concentration for the 0.125 mm sample than in normal atmospheric conditions. The same behaviour is observed for two data sets of 0.61 bar and 30% oxygen concentration for the same 0.125 mm sample. Nonetheless, the opposite behaviour is exhibited by the 0.25 mm sample, where the longest ignition delay time occurs at 0.7 bar and 30% oxygen concentration. This change of regimes coincidently also occurs for flame lengths (will be discussed later), thus strongly suggesting that such changes might happen in the gas phase. Olson [9] reported that there was no obvious dependency of ignition delay in normoxic environments (when increasing oxygen and decreasing pressure) during microgravity experiments on a thermally-thin cellulosic sample. The only notorious difference with the previous study is regarding the PDMS and the formation and transport of silica.



Figure 3 – Ignition delay time in normoxic conditions for two PDMS samples as a function of the oxygen concentration (top panel) and ambient pressure (bottom panel). Data from 0.61 bar and from 30% are also plotted for comparison.

As described earlier, during the combustion of siloxanes, one of the by-products is silica ash or SiO_2 that can be formed during the gas-phase and solid-phase [14,15]. Oxygen variations can affect the formation of silica-ash since more oxidizer is readily available to form silica ash. Thomas and Kendrick [27] reported higher activation energy for depolymerisation in vacuum conditions when compared to a thermo-oxidative environment. Concerning the transport of the ash, a reduction in pressure decreases the buoyant forces and more silica- might accumulate on the surface. However, it is not clear how the formation of silica and its transport is effectively affected by the environmental conditions and how the piloted ignition is affected.

3.2. Flame spread behaviour

The flame spread rate results as a function of the oxygen concentration and ambient pressure are plotted in *Figure 4*. The flame spread rates under different fixed

ambient pressures exhibit a linear dependency on the oxygen concentration. As the oxygen concentration is increased, the flame temperature increases due to chemistry [28,29]. In turn, the heat from the solid-phase to the gas-phase increases. Then the increase in flame temperature, and increase in gas-phase properties, is directly proportional to the flame spread rate, as confirmed by $V_f = \frac{k_g \rho_g c_p (T_f - T_p)}{\rho_s c_s \tau (T_p - T_o)}$. In addition, a reduction of the flame temperature translates into a decreased in the induced buoyant velocity.

The pressure shows to affect the flame spread differently, as two apparent regimes emerged, see *Figure 4* (bottom panel). As the pressure is reduced, the flame spread rates decrease linearly with decreasing ambient pressure for any fixed oxygen concentration. This dependency is showed to be weak, as compared to the oxygen concentration dependency. If the ambient pressure is reduced further, the flame spread rate decreases in an asymptotic manner, reaching a limiting vertical value. Such behaviour has been reported previously with different materials [30–34].

As pressure is reduced, the incident heat flux from the flame is reduced. The heat from the gas-phase to the solid-phase is reduced proportionally as the flame thickness is increased with decreasing pressure [31]. The flame spread has a weak dependence on the pressure but is heat transfer controlled [32]. For the nonlinear and asymptotic behaviour, the gas-phase diffusion is inversely proportional to the pressure $(\alpha \sim 1/p)$. In contrast, the reaction time increases with decreasing pressure. Thus, flame spread rate is controlled via kinetics for the asymptotic regime (Damköhler number becomes small). It has been hypothesized that at very low pressure, there is a combination effect of radiative losses (via solid-phase) along with a decrease in kinetics [35]. Bhattacharjee et al. [35] have shown that a radiative parameter is proportional to $(1/p^2g)^{2/3}$ and the Damköhler number is proportional to $(p/g)^{2/3}$.





Figure 4 – Opposed flame spread rate as a function of the oxygen concentration (top panel) and ambient pressure (bottom panel) for a 0.125 mm thick PDMS sample.

The flame spread rates for normoxic conditions as a function of the oxygen concentration and ambient pressure are plotted in Figure 5. A combined effect of reducing ambient pressure while increasing the oxygen concentration has mainly consequences via heat transfer. As seen previously, the increase of oxygen results in higher flame temperatures. On the contrary, reducing pressure enlarges the flame (thick optical flame) and reduces the heat transfer from the flame to the solid at the leading edge. The results indicate that heat transfer via chemical reaction has a larger effect on the flame spread rates, at least away from extinction conditions. Also, the flame spread rates are proportional to the sample thickness as expected. Thus, normoxic conditions do not offer an improved flammability scenario for PDMS. Similar results have been reported for cellulosic fuels [36].





Figure 5 – Opposed flame spread rates as a function of the normoxic condition for two PDMS samples (thicknesses of 0.125 mm and 0.25 mm).

The flame length results are depicted as a function of oxygen concentration and ambient pressure In Figure 6. The dependency of flame length on the oxygen concentration is exponential for all data sets. Bhattacharjee et al. [37] derived an expression for the flame length in the thermal regime $L_f \sim 0.0345[(T_f - T_v)/((T_p - T_\infty)y_{0_2})]^2 L_g$. For all the pressures tested, it was assumed that the thermal regime will hold away from extinction. In the former equation, the flame length is directly proportional to the square of the oxygen concentration and flame temperature. Thus, it coincides with the current results. The effect of the oxidiser on the flame length relies on two aspects, the supply of fuel and the local supply of oxidiser. Increase in oxygen results in increasing flame temperature and soot oxidation [38], which in turn increases the heat feedback (radiation) and elongates the flame. Under buoyant conditions, the buoyant induced flows do not affect the supply of an oxidiser, and thus flame lengths will be mainly dependent on the oxygen concentration.

The flame length dependency on ambient pressure exhibits a non-monotonic behaviour, as seen in Figure 6 (bottom panel). For the increasing regime in flame length with respect to the increasing pressure, similar results have been reported in the literature [39,40]. As flame height is defined by the supply of fuel and oxygen entrainment in the combustion zone, these would also be affected by changes in ambient pressure. Increasing pressure while fixing oxygen concentration affects the local supply of oxidiser since the induced buoyant velocity is proportional to pressure. At the same time, an increase in pressure increases the flame temperature, providing more heat feedback to increase the fuel supply rate [39,40]. Consequently, the leading mechanism is fuel supply via thermal transfer as the flame length increases with increasing pressure.

For the second regime, where flame lengths decrease with increasing pressure, increasing pressure leads to an excess of fuel which will be burnt in the flame region. The flame height or length is determined by the ratio of the heat of gasification to the stoichiometric oxygenfuel mass ratio [41]. For increased pressure, there is an excess of fuel supply (infinitely large Damköhler number). Then, the dominant force is the oxidiser supply because the induced buoyant velocities increase with increasing ambient pressure. Thus, the local oxidiser supply increases the stoichiometric oxygen-fuel mass ratio, and the flame length decreases, as suggested. Another possible mechanism that might aid in the local oxygen supply is pressure-induced turbulences. Ban et al. [42] also reported flame height (gaseous diffusion flame) decreasing with increasing pressure. They attributed it to the shift from a laminar regime towards a turbulent regime.



Figure 6 – Flame length as a function of the oxygen concentration (top panel) and ambient pressure (bottom panel) for a range of oxygen concentrations for a 0.125 mm thick PDMS sample.

The corresponding flame length results in the normoxic environment are plotted in *Figure 7*. From the combined effect of oxygen concentration and ambient pressure, the former seems to help in providing a large fuel supply and large flames. Thus, oxygen supply dominates the flame lengths under normoxic conditions. The flame length is nearly the same for both sample thicknesses, and this indicates that there is no substantial increase in fuel supply from a 0.125 mm to a 0.25 mm thick PDMS sample. It appears that oxygen supply dominates the flame lengths independently of sample thickness for thermally-thin fuels, at least in the current investigation.



Figure 7 – Opposed flame lengths as function of the normoxic condition for two PDMS samples (thicknesses of 0.125 mm and 0.25 mm).

The flame length results are plotted against the oxygen partial pressure in Figure 7. As seen in the top panel, for normoxic conditions to be kept (vertical line), the maximum flame length occurs at 0.61 bar. In the bottom panel of Figure 8, two regimes in flame length are observed as a function of the oxygen partial pressure. The exact mechanism behind both regimes that was described earlier applies in Figure 8. What is interesting is the change of regimes that occur at the actual normoxic condition. Thus, normoxic conditions help flame spread and provide the best conditions for the longest flame lengths in buoyant scenarios. In reduced gravity, such behaviour might not be necessarily the same, and it thus worth further investigation.



Figure 8 – Opposed flame length as a function of the Oxygen partial pressure (Opp). The vertical line indicates the normoxic condition. The sample was 0.125 mm thick.

3.3. Flammability map

Figure 9 shows the boundary limits as a function of the oxygen concentration, ambient pressure, and partial pressure. These extinction limits resemble the radiative branch found in microgravity. In normal-gravity, a similar extinction boundary has been found with other materials [32,43]. Previous studies have suggested that the extinction limits at low pressure are dominated by kinetics. Molecular diffusion is inversely proportional to the ambient pressure [32], and that the reaction order decreases with decreasing pressure [44]. Thus, the flow time is short, and the chemical time is very low, yielding a smaller Damköhler number. Also, as the pressure is reduced, the heat losses increase and can also contribute to extinction [45]. A recent numerical study suggested that there is an interaction between kinetics and heat losses at low pressure [35]. Thus, the extinction conditions at low pressure might be a combination of a reduction in heat losses through the solid-phase and gas-phase and a combination of slower chemical reaction rates.

The normoxic curve and the hypoxic limit are also plotted in *Figure 9*. As seen, between those limits, the PDMS still encounters favourable flammability conditions. This zone corresponds to the earlier flame spread and flame length results, which were enhanced in normoxic conditions. Thus, this particular PDMS is still flammable in normal gravity scenarios. It is not clear how different these limits would be in microgravity conditions. In addition, it is not clear what it is the contribution of the silica-ash deposition, if there is any, at low pressures.



Figure 9 – Flammability limits for a range of ambient pressures and oxygen concentrations. The normoxic and the hypoxic limit are also shown. The data are for opposed flame spread over a 0.125 mm thick PDMS sample.

4. CONCLUSION & FURTHER WORK

The experimental investigation has shown that PDMS membranes have a specific flammability behaviour and striking phenomena for a range of environmental parameters. The main findings are summarised in Table 1.

Topic	Parameter	Dependency	Dominant mechanism	Observation
Ignition	O_2	Linear-like	Chemistry	
	р	Linear-like	Chemistry	
	Noxic	Non-monotonic	Not established	Dependency is reversed for sample thickness.
Flame spread	O ₂	Linear	Chemistry/thermal transfer	
	p –	Linear	Thermal transfer	
		Asymptotic	Thermal transfer & Kinetics (?)	
	Noxic	Linear	Chemistry (O ₂) dominates over losses (due to p)	heat

			I O 1 1 C 1 1		
Flame length	O_2	Exponential	Increase O_2 supply and fuel supply (due to heat transfer) & increase soot		
		Exponential	(due to heat transfer), & merease soot		
	р	$d \downarrow $ Non-tonic Non-tonic	Fuel supply via thermal transfer + mixing with O_2 (\uparrow induced buoyant		
			velocity)		
			with O_2 (\uparrow induced buoyant velocity and turbulences)		
	Noxic	Linear	O ₂ dominates over p	Almost independent fuel thickness	on
Near- limit	O ₂ & p	Asymptotic At low p	Thermal transfer (\uparrow losses) & reduced kinetics (?)		
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Table 1 – Resulting dominant dependencies and corresponding dominant mechanisms as a function of various parameters. The following abbreviations are used: O_2 (oxygen concentration), p (ambient pressure), Noxic (Normoxic), \uparrow (increasing) and \downarrow (decreasing).

The largest flame length occurred at the change of regimes and close to the normoxic condition. In addition, ignition and flame spread can occur more readily in normoxic situations since the effect of increasing oxygen is greater than that of reducing ambient pressure. From the extinction maps, it is clear that flammability is viable for regions between the normoxic and hypoxic limits. A normoxic environment might increase the flammability of PDMS materials on Spacecraft and could compromise fire safety. As such, these preliminary results should be considered for future environmental spacecraft design.

Most of the mechanisms behind the dependencies are somewhat straightforward. However, some dependencies remain ambiguous or still require further investigation. These unknowns are the combined effect of heat losses and reduced kinetics at low pressure for flame spread and near-limit, environmental conditions on the silica-ash formation and transport, and the impact of normoxic conditions on the flammability behaviour. These unresolved phenomena require further investigation in normal gravity and microgravity environments.

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