An investigation of LNG permeability within perlite insulation

Steven Betteridge, Senior Technical Safety Engineer, Shell Research Ltd, Shell Centre, London SE1 7NA

In recent years, there has been an increasing demand for Liquefied Natural Gas (LNG) as a clean and efficient fuel for commercial vehicles such as trucks and ferries. Next to liquefaction plants and regasification terminals, this distribution chain also has retail fuel stations and bunker terminals that can be located relatively close to urban environments. The vacuum insulated pressure vessels used to store LNG have been used safely within the LNG industry for over two decades, however as the number of tanks and their associated volumes increase, it is important to demonstrate the vessel's integrity across its whole lifespan. This is made more difficult because the inner vessel and associated pipework and fittings in the annulus cannot not be accessed during routine maintenance due to the use of expanded perlite or super insulation to improve the thermal insulation. This not only requires better mechanical designs to provide the required assurance, but also to consider how leaks from the annulus space could be mitigated, even if these leaks are considered to be very low frequency events. Such mitigation may lead to more costly designs if LNG can be demonstrated to permeate through the annulus insulation and collect at the base of the outer tank, for instance using stainless steel to construct the outer tank.

To understand the consequences of a leak in the annulus space, Shell investigated the permeation of LNG and gas through expanded perlite in a medium sized experimental rig that was designed to simulate the design of a double skinned pressurised vessel. In these experiments, a 300 mm layer of expanded perlite was compacted into a 10" pipe to a density of 90-110 kg m^{-3} to simulate typical design scenarios for larger vacuum insulated vessels. To measure the permeation of both liquid and gas through the perlitree, a series of thermocouples were placed within the column of perlite and any gas that did permeate was collected and measured. In addition, pressure sensors were placed above and below the perlite surface to both quantify the reservoir conditions and determine the pressure drop across the perlite. This paper will show the results from these experiments; understanding whether a sudden release of cryogenic LNG causes the perlite surface to break up and allow the fluid to permeate more easily and quantifying the rate of gas and liquid permeation through the perlite.

Keywords: LNG; Perlite; Permeability; Hazard; Catastrophic Failure; Double Wall Insulated Vessel

Introduction

The storage of Liquefied Natural Gas (LNG) at liquefication trains and import terminals has typically been within large atmospheric tanks with concrete secondary containment. However, in the last couple of decades the commercial use of LNG has expanded in response to concerns about the use of cleaner and more efficient fuels. These operations are often at a smaller scale and closer to the public than previous cryogenic storage and as a consequence the LNG has been stored in double-skinned pressurised vessels. Not only does this aid the transfer of LNG product for some operations, but also reduces natural boil off.

The boil off of cryogenic fluids not only has a direct impact on the cost effectiveness of the operation, but also affects the criteria for safe operation via the design choices for the maximum working pressure for the inner vessel and if/how boil off gases are vented to atmosphere. Consequently, the boil off is minimised by insulating the annulus region between the inner and outer wall of the tanks with an insulating material as well as evacuating the remaining air to create a partial vacuum. For small pressurised cryogenic vessels, this material is typically a multi-layer metal foil insulation. Alternatively, and especially for larger vessels, the annulus space is filled with expanded perlite. Perlite is an industrial mineral of amorphous volcanic silicate glass and its good thermal insulating properties are obtained by heating it to 1000°C, which causes water within the perlite to vaporise instantly and the perlite "pops"; expanding up to 20× its original volume. The benefits of a powder insulation, such as perlite, for improving the thermal performance of cryogenic tanks is well established (Fulk 1959) and investigations to demonstrate its thermal conductivity and hence impact on boiloff as a function of density, vacuum pressure etc. have optimised the design of tanks using perlite (Kropschott 1963). Historically this has been used for commercial storage of cryogenic fluids, such as liquid hydrogen at NASA sites (Krenn 2012), or at the sites of cryogenic suppliers, where larger quantities of cryogenic fluids are stored for long periods. Consequently, there was a natural progression to use perlite for pressurised LNG vessels and they have been used safely within the LNG industry for over two decades. However, as the number of tanks and their associated volumes increase, it is important to demonstrate the vessel's integrity across its whole lifespan.

In Shell, a Development Release was written in 2015 to standardise the deployment of double skinned, vacuum insulated LNG tanks. The development release was based upon ASME VIII division 1 design and included cold stretching of the inner vessel to allow for higher stresses. This option was partly selected to provide additional assurance on the integrity of the inner tank, because the inner vessel and associated pipework and fittings in the annulus cannot not be accessed during routine maintenance due to the use of expanded perlite. Good engineering design and controls during construction should therefore ensure that the likelihood of a failure at the inner vessel should be minimised. Never the less, it is also important to consider how leaks from the annulus space could be mitigated, even if these leaks are considered to be very low frequency events. For instance, if LNG can be demonstrated to permeate through the annulus insulation and collect at the base of the outer tank, then it may be necessary to construct the outer tank using stainless steel to avoid brittle fracture, which would significantly increase the overall cost of the tanks.

Consequently, Shell investigated the permeation of liquid and gaseous natural gas through expanded perlite to gain further understanding if there was a leak of LNG in the annulus space. This paper reports on the outcome from eight experiments in a medium sized experimental rig. The experimental rig used a 300 mm perlite layer within an instrumented 10" pipe to simulate equivalent designs for double skinned pressurised vessels that were being considered for a number of Shell projects.

The paper will show the results from these experiments; providing greater understanding whether a sudden release of cryogenic LNG causes the perlite surface to break up and allow the fluid to permeate more easily, as well as quantifying the rate of gas and liquid permeation through the perlite, and hence provides guidance for future designs of double skinned pressurised vessels that using perlite.

Experimental Setup

It is not practical to simulate an LNG release from an actual vacuum insulated LNG vessel. Therefore, a medium sized experimental rig was constructed to simulate the equivalent design of a double skinned pressurised vessel, as shown in Figure 1 . In these experiments, the layer of expanded perlite was confined within a 10" stainless steel pipe and the LNG was applied to the top surface, while the bottom surface was connected to a collection vessel to measure the quantity of gas that permeated through the perlite. In all experiments, a 300 mm layer of expanded perlite was packed to achieve an overall density of 94 kg m^{-3}. Both the depth and density of the perlite were chosen to match the specification that had been proposed for a 1000 m^3 LNG storage tank at a new LNG terminal. It should be noted that in all the tests, the perlite was not under a vacuum and therefore thermal conduction was higher than would be expected during normal operation of a vacuum insulated tank. This is not an unreasonable assumption, because the vacuum is normally assumed to be quickly lost if there is a loss of containment.

Figure 1: Experimental rig for Test 2. The outer stainless steel surface is lagged to improve the thermal insulation

The experimental setup was adapted during project to test different aspects of LNG permeation. In Tests 1-5, the LNG was supplied to the rig at saturation conditions and with a small pressure above atmospheric conditions through a diffuser to reduce losses as the top section of the rig cooled down. Once the rig cooled below -160°C, LNG started to accumulate on top of the perlite surface as measured by a thermocouple that was situated just above the perlite surface (T2). Thermocouples T3 and T4 were positioned approximately 100 mm and 200 mm respectively below the upper surface of perlite, approximately 50 mm from the side wall. Thermocouple T5 was placed 10 mm above the base of the perlite along the same line. A 6^{th} thermocouple was added to later experiments at the base of the perlite and near the orifice. The pressure in the rig was measured in two places; P1 in the upper chamber, where LNG was injected and P2 adjacent to T5 under the perlite. Note that P2 was in a short tube connected to the base of the perlite and so was not directly affected by mechanical pressure applied to the perlite.

Gas was allowed to permeate through the bottom layer of the perlite through either a 5 mm orifice within the base plate (Tests 1 and 2) or a 5" orifice connected to a collection chamber (Tests 3,4 and 5). In all five tests, any gas that escaped through the bottom plate was directed though a stainless steel tube and a manually operated valve into a 200 litre collection chamber. The collection chamber was prefilled with water and therefore the permeated gas could be measured through the displacement of water. The diameter of the stainless steel tube was initially 10 mm but was replaced and increased to a pipe with an internal diameter of 22 mm for Tests 4 and 5 in case the gas outflow was limited by the pipe bore. The configuration for Tests 3,4 and 5 using the 5" orifice below the perlite layer is shown in **Figure 2**.

The experimental arrangement in the first five tests was designed to make it easier to insert and pack the perlite. However, the perlite was not pre-cooled before the LNG was added, resulting in both cooling and permeation occurring at the same time in the perlite layer. This was not representative of the physical setup in a vacuum insulated tank, where the perlite in contact with the inner tank would be pre-cooled to temperatures close to -160°C before any loss of containment. Therefore, in Tests 6-8 the experimental rig was modified; a cryogenic chamber, separated from the perlite by a stainless steel plate, was pre-cooled using liquid Nitrogen (LIN) to allow the perlite to cool to a temperature just above -160°C. The LNG was then released into the perlite chamber through an 5-20 mm orifice in the stainless place via a remotely operated valve. This modification also allowed the effect of a jet release of LNG onto the perlite surface to be investigated.

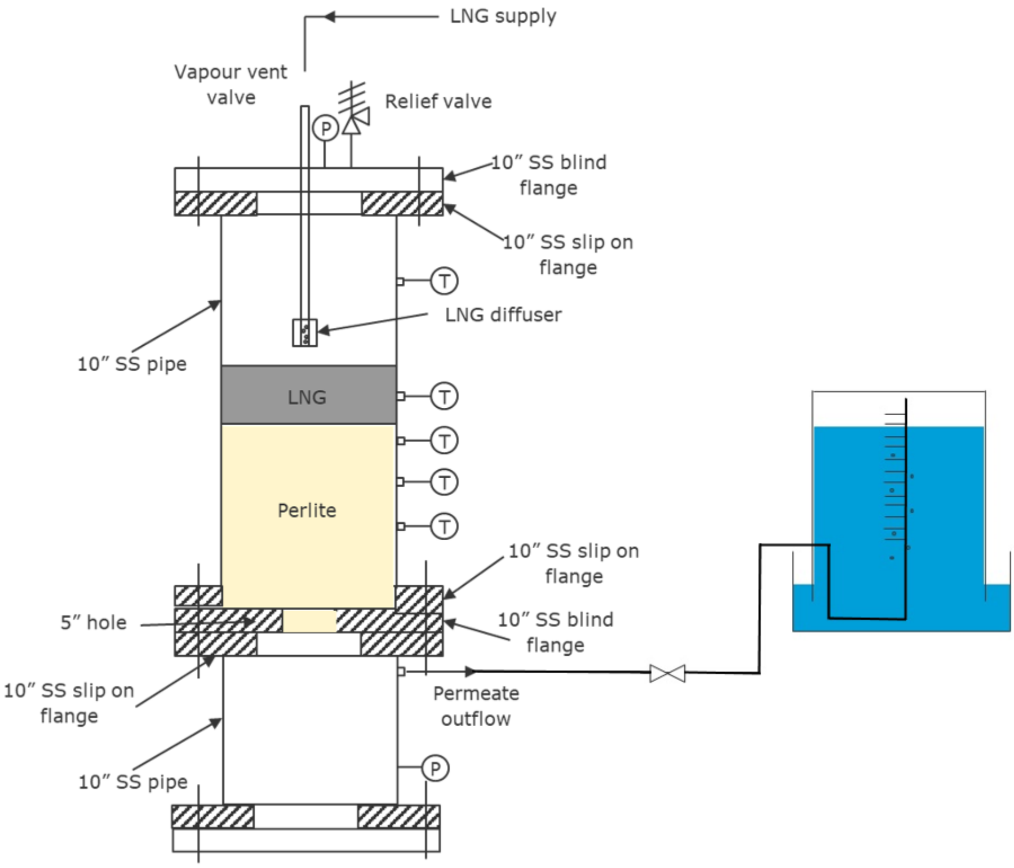

Figure 2: A schematic of the experimental test rig used for experiments 3,4 and 5, where the LNG is released directly on top of the expanded perlite. This shows a 5" orifice below the perlite, with a chamber to collect any perlite that was lost. The thermocouples were labelled T1 – T5, starting with T1 in the vapour space and T5 at the bottom of the perlite layer.

A schematic of the experimental test rig used in Tests 6-8 is shown in **Figure 3**. The LNG was released through a 1" orifice in this plate and then through a 10 mm thick plate attached below the 15 mm stainless steel plate, which effectively protruded into the perlite layer. The 10 mm plate was replaced for each experiment to allow a different hole size (5 – 20 mm) to be used in each experiment. The perlite was held in position using a wire mesh (Size: 500 Mesh - 27 microns aperture). To provide additional strength, and stop the perlite bending the mesh, the mesh was positioned on a 1.5 mm stainless steel plate with many 1" holes drilled into it. The pressure sensor P1 was positioned in the LNG feedline, while P2 was positioned in the chamber under the mesh. The position of the thermocouples are given in **Figure 3**.

In the final test, Test 8, an additional pressure sensor, labelled P2, and temperature sensor, labelled T10, were added and positioned 30 mm below the orifice within the perlite. This position corresponded to the location where a void formed in Tests 6 and 7. In this final test the pressure sensor in the collection chamber was labelled P3. To ensure that LNG flowed into the perlite during the experiment, the top valve was closed, and the side valve was kept open until liquid started to appear. This side valve was then closed, and once the correct pressure had been reached, the LNG was released into the perlite layer via a remotely operated valve.

During each experiment, the times for the following events were all recorded;

- When cooling started,
- When LNG flowed into the perlite,
- When the bottom valve to the gas collection vessel was opened
- When gas started to be collected

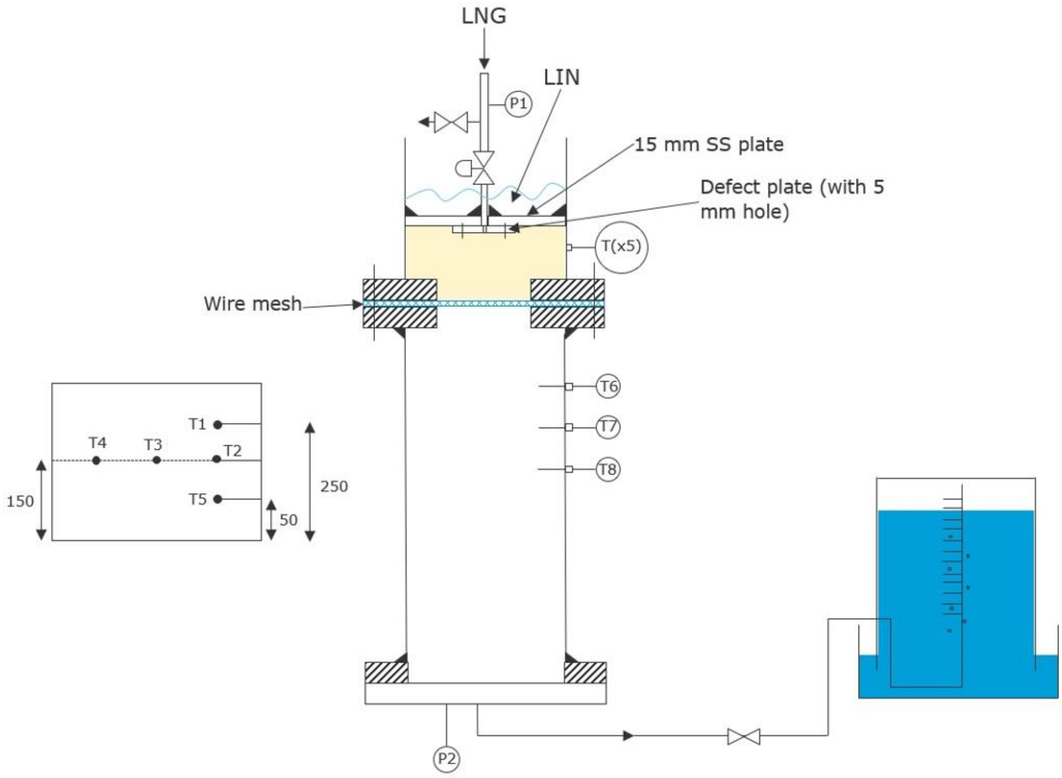

Figure 3: A schematic of the experimental test rig for experiments 6,7 and 8. In this arrangement the perlite can be pre-cooled before the LNG is released through a small orifice in the 15 mm stainless-steel plate. A wire mesh is used to hold the perlite in position. The thermocouples within the perlite layer were positioned as shown in the insert.

At the end of each experiment, the experimental rig was purged with nitrogen gas for 24 hours, before it was opened to determine the state of the perlite. This was specifically done to see if a leak path had formed through or along the edge adjacent to the inner wall of the 10" pipe. This was achieved by visual inspection and using an industrial vacuum hose to suck up the perlite to reach the bottom layers. The use of an industrial vacuum hose was required to limit exposure to the perlite dust and because it would have required a hand towel to break up the hard packed perlite. One benefit of this approach was to check the position of the thermocouples at the end of test in case the packing had displaced their original positions. In general, the position of the thermocouples was found to remain within ± 10 mm of their intended position. The mass of perlite that fell into the collection chamber was also quantified at the end of Tests 3, 4 and 5.

Expanded perlite

The same grade of expanded perlite as that used by a tank manufacturer for large LNG tanks was obtained and used in all eight tests. The supplier datasheet for this perlite showed that it had a fairly typical composition > 66% SiO_2, and < 18% Al_2O_3, < 3% Fe_2O_3, < 5 %CaO + MgO and < 8% Na_2O + K_2O. The density of this perlite before compaction was measured to be about 48 kg m^{-3} and hence was within the range of 45 – 65 kg m^{-3} quoted on the datasheet. On the datasheet an approximate particle size distribution was also provided, this indicated that less than 10% of the grains were greater than 0.5 mm, while at least 40% of the perlite grains were smaller than 0.2 mm.

Packing of perlite

The process of packing perlite into double skinned tanks is considered proprietary by the tank manufacturers. However, the process is generally believed to include (Šterba 2015):

- the use of a partial vacuum to suck the perlite into the annulus space between the inner and outer walls
- the use of an eccentric motors to vibrate the outer tank wall to settle the perlite
- preheating the perlite to reduce the water content

The volume of the annulus space and the mass of perlite is then used to determine the average density that has been packed into the double skinned tank. It was not possible to achieve the same density increase (48 kg m^{-3} to 94 kg m^{-3}) within these tests using different combinations of the techniques above, even when leaving the vacuum running overnight. This was surprising considering the relative simple geometry of the 10" pipe when compared to the complex geometry in a double skinned tank with the associated pipework and fittings in the annulus space. Consequently, to achieve the required density a static load was used, in conjunction with the partial vacuum and vibration motor, to increase the density from approximately 75 kg m^{-3} to the required density. The final density in all eight tests was calculated to be 94 ± 1 kg m^{-3}.

Results

A matrix showing a high level summary of the different tests is shown in **Table 1**.

Test	LNG feed	Pressure (barg)	Comments
1	Direct onto Perlite	2.2	Initial test
2	Direct onto Perlite	1.7	Duplicate Test 1 at reduced pressure
3	Direct onto Perlite	1.4	5" downstream orifice + perlite collection
4	Direct onto Perlite	3.2	Duplicate Test 3 at higher pressure
5	Direct onto Perlite	1.3	Repeat - more thermocouples in horizontal plane at 100 mm
6	Via a 5 mm orifice	5	LNG release after precooling perlite
7	Via a 20 mm orifice	3.7	Repeat Test 6 with larger orifice
8	Via a 10 mm orifice	0.5-8	Ramping pressure up slowly and cycle pressure between 0.5 and 8 barg

Table 1: High level summary of experimental configuration

To illustrate many of the common features seen during each of the first five tests, the temperature and pressure sensors measurements recorded during Test 5 are shown in **Figure 4**. In each of these experiments, the start time was defined to occur when LNG was released into the top chamber shown in **Figure 2**, and as expected the pressure instantly increases to 2 barg as the initial LNG is vaporised as it cools the chamber and top surface of perlite. As illustrated by these results, in each experiment it took about 1000 s for the chamber to reach -150°C and hence for liquid to start to accumulate on the perlite surface. Once liquid started to accumulate, both the LNG supply and the relief valve were adjusted to manage the internal pressure to achieve the specified test pressure. Subsequently, it often took about 1 hour to achieve the requested pressure and also have confidence about the internal process conditions. For instance, in Test 5 the pressure reached the specified pressure (1.5-1.6 barg) after 2500 s, but the temperature of the vapour space was too warm (-70°C) and therefore the bottom valve was not opened for a further 2000 s. During this time the perlite gradually cooled to LNG temperatures, although the time did vary in each experiment and during each experiment it was not clear if the temperature reduction was due to liquid or gas permeation or simply thermal conduction.

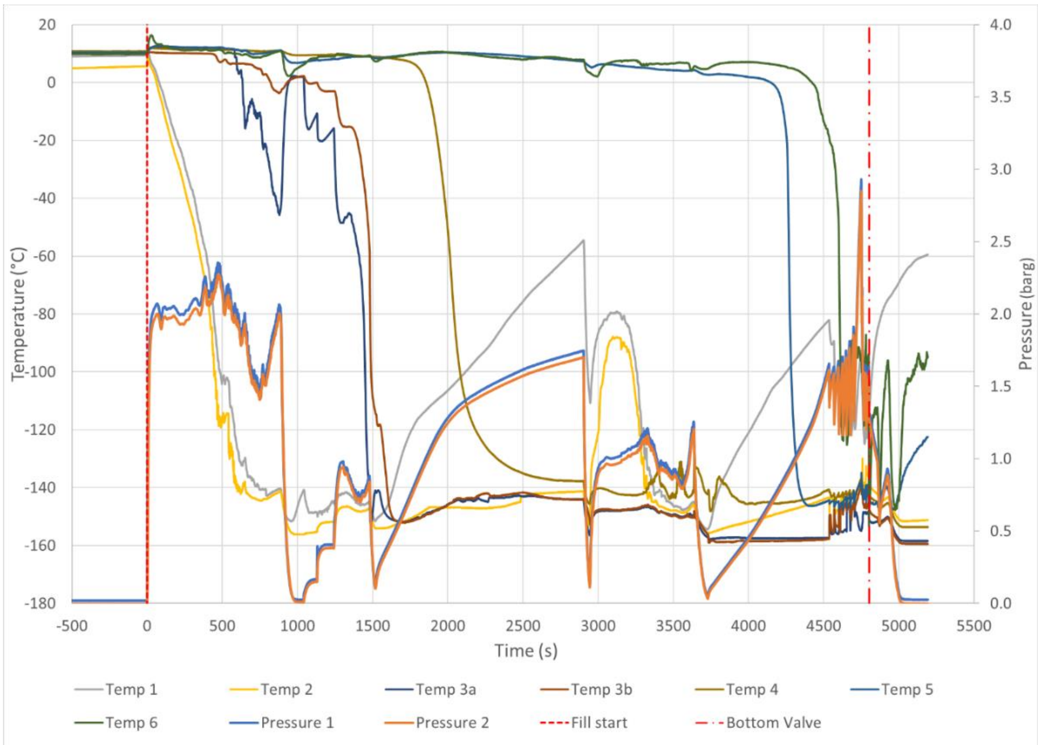

Figure 4: Measured temperature and pressure values recorded during Test 5

The results from the first five tests indicated that the pressure above and below the perlite increased as soon as filling started. More importantly, the pressure below the perlite tracked the pressure in the vapour space above the perlite throughout each of these experiments. It is postulated that the pressure could equalise through lower density regions at the interface of the perlite and the 10” pipe wall. Once the bottom valve was opened, there was an immediate reduction in pressure above and below the perlite layer. At the same time, gas was collected in the external vessel, resulting in the displacement of 200 litres of water in this vessel within 40-90 s.

When the vessel was opened at the end of the test, a void space adjacent to the 10" pipe wall was clearly observed in Tests 2, 4 and 5, as shown in **Figure 5**. A void was also seen in Test 1, but it is speculated that this was generated, or at least enlarged during the degassing operation after the experiment finished. In subsequent tests a more gentle degassing process was used. The void in these tests extended to the bottom surface and therefore provided an easy pathway for the liquid to pass to the bottom chamber. The thermocouple results suggest that this void was generated when the bottom valve was opened and hence when there was suddenly a large pressure difference between the top and bottom chambers.

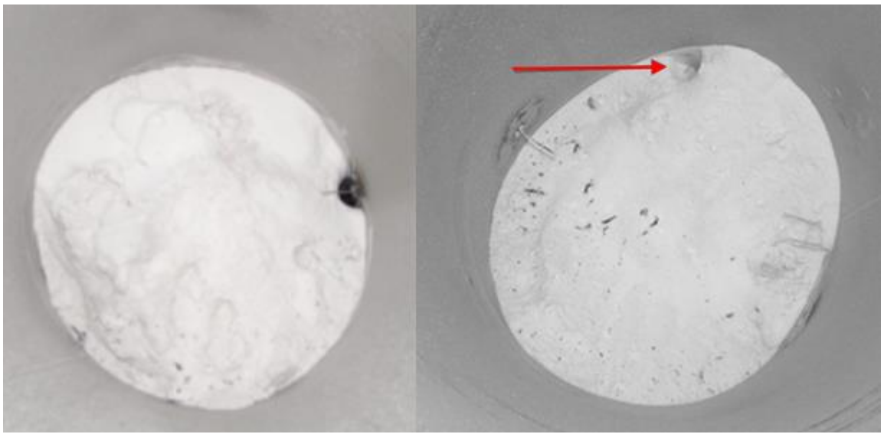

Figure 5: The void created adjacent to the pipe wall in Test 4 (left) and Test 5 indicated by arrow (right)

Perlite was also collected in the bottom chamber when the 5" orifice was used under the perlite layer in Tests 3, 4 and 5. This was a relatively small amount in Test 3 (51 g), when no void was generated along the wall, but approximately 200 g was lost in Tests 4 and 5 and this is illustrated for Test 4 in **Figure 6**.

Figure 6: Perlite collected (left) and the bottom surface of perlite (right) after completing Test 4. The perlite void extended approximately 100 mm into the chamber

In the last three tests, Tests 6-8, the experimental arrangement was modified so that the perlite could be cooled to cryogenic temperatures before the release of LNG and the LNG was released through a small circular orifice – see **Figure 3**. As expected, the results were significantly different, which can be seen in **Figure 7** for Test 7, where the LNG was released through a 20 mm orifice. As can be seen from the time axis, the perlite was cooled for over 2500 s using liquid nitrogen, before the top valve was opened. In this time the thermocouple at a depth of 50 mm, reached a temperature of -80°C in all three tests. To ensure that only liquid exited the orifice, a side valve was opened to pre-cool the pipework. The top valve was opened once liquid was exiting the side valve and the required pressure of the LNG had been achieved in the reservoir.

Contrary to the first five tests, the results showed no pressure increase under the perlite, once the top valve had been opened. In Test 7, the pressure in the bottom chamber did register a maximum pressure of 1 barg once the bottom valve to the collection vessel had been opened, but this was always at least 0.5 barg lower than the top pressure. In Test 8, which used a 10 mm orifice, the maximum pressure in the bottom chamber was never greater than 0.5 barg and in Test 6, which used a 5 mm orifice, there was no noticeable pressure change in the bottom chamber. These results showed that pressure equalisation seen in Tests 1-5 was due to the setup of the experiment and in reality the pressure experienced by the outer tank wall would be lower than the inner pressure.

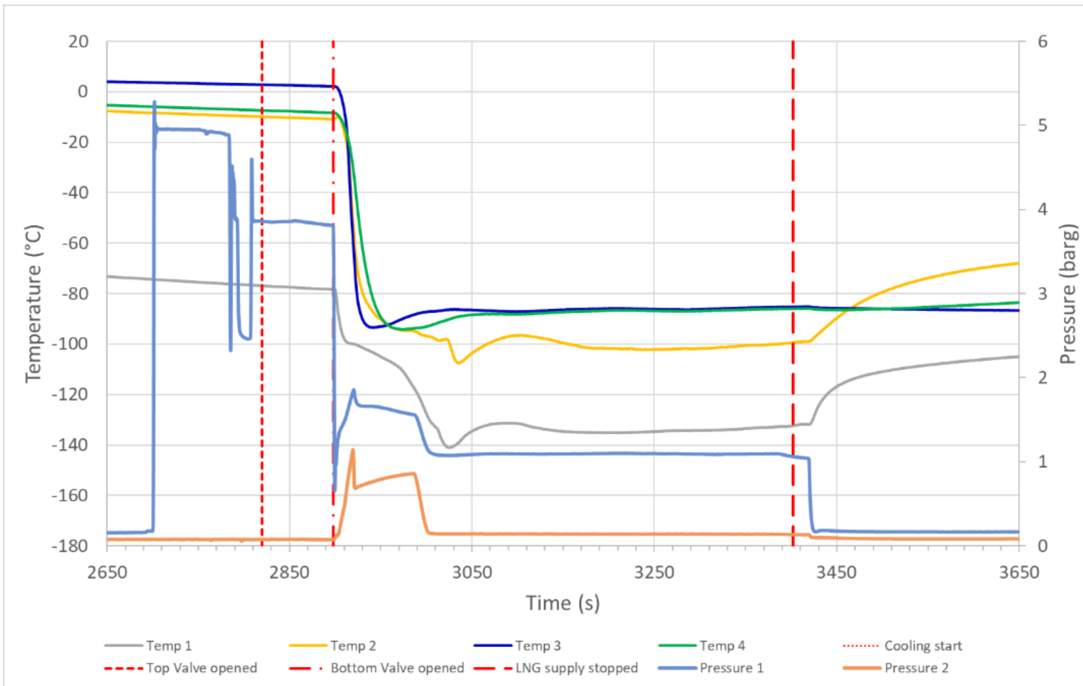

Figure 7: Measured temperature and pressure values recorded during Test 7, where the LNG was released through a 20 mm orifice

There was an immediate reduction in temperature for thermocouples situated at a depth of 150 and 250 mm in Test 7, although they did not record temperatures lower than approximately -100°C. In Test 8, the thermocouples did register temperatures at -160°C, but they appeared to respond more similarly to the change shown in Test 5, such that there was a more rapid change in temperature after a period of time. In this case T3 and T4 showed a rapid change 300 and 500 s after the top valve was opened, whereas T2 at the same depth (150 mm) took 1000 s. T5, at a depth of 250 mm, showed a slow temperature drop for 2500 s after the top valve opened and then reduced over 500 s to -150°C. More significantly these temperature reductions did not appear to be affected either when the LNG pressure increased to 8 barg and the bottom valve was opened twice. In Test 6, the thermocouples at a depth of 150 mm also reached -160°C, but only after the top valve had been open for approximately 2 hours. In addition, the thermocouple at a depth of 250 mm only reached -40°C after 4 hours.

The most significant result from the final three tests was the effect of orifice size on the amount of gas collected. Only in Test 7, was gas collected as soon as the bottom valve was opened; resulting in 200 litre of water being displaced within 50 s. However, no gas was collected in Test 6 even after leaving the top valve open for 4 hours, while in Test 8, gas was only collected after the input LNG pressure had been raised to greater than 5.5 barg. The gas collection stopped once the LNG pressure reduced back to 1 barg. Only when the bottom valve was opened for the 3rd time, did gas collection occur at a relatively low pressure (1 barg) and then the rate of gas flow increased after the valve had been left open for 100 s, which may correlate to an increase in the downstream pressure, when the pressure increased from 0.1 to 0.4 barg.

When the chamber was opened at the end of the experiment, no void was seen along the outer wall of the pipe. However, a void had been created in the top surface of the perlite by the LNG jet in Test 6 and 7, as can be seen in **Figure 8**. In Test 6, the void was measured to be approximately 60 x 80 mm and be 140 mm deep, whereas in Test 7, the void had a larger area 140 x 80 mm, but only extended 30 mm. It is presumed that the perlite that occupied this volume was squashed into the bulk of the perlite, although there was no obvious visual change to the perlite at the edge of this void. Some attempts were made to quantify the density of the perlite along the edge of the void, but the perlite was too fragile and broke into fine powder as it was handled. Somewhat surprisingly no void was observed in Test 8, which used an orifice of 10 mm, although the perlite did appear to be less compacted near the orifice exit.

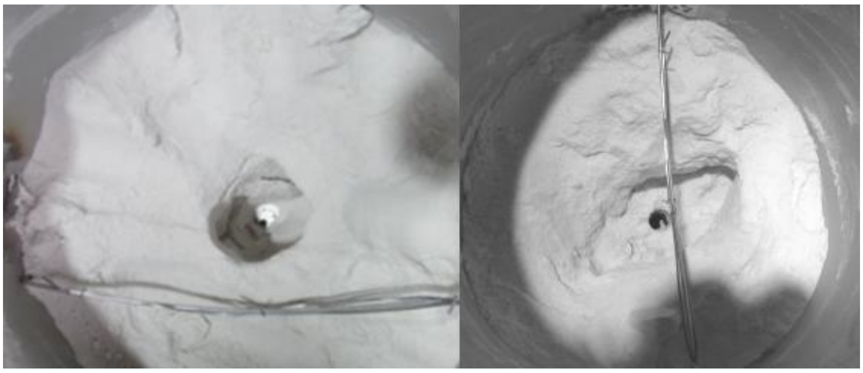

Figure 8: The void formed at the top surface of the perlite by the LNG jet in Test 6 (left) and Test 7 (right)

Discussion

Void formation

During the design of these experiments it was postulated that gas and liquid permeation was more likely to occur at the interface with metal surface, since there would be reduced friction (compared with the bulk perlite) and the packing process may mean that it is difficult to achieve the same packing density in these regions. This has been seen in the industry even for relatively simple designs, e.g. a sphere, where regions of lower density perlite have formed over time and caused an increase in the overall boil off (Krenn 2012).

In practise, the formation of a void within the perlite was seen in most of these experiments; whether it was a small gap adjacent to the edge of the 10” pipe wall, loss of perlite from the bottom surface through the 5” orifice or created in the vicinity of the 5-20 mm orifice. This shows that the pressure of an LNG release can obviously modify the packing of the perlite granules and create a pathway that relieves the pressure. Therefore, some void formation might be expected to occur during an actual failure in to the annulus space of a double skinned tank. However, further quantification of when/how these voids were generated was difficult in these experiments because the voids were only analysed at the end of each experiment.

In the first five experiments, the LNG was placed in direct contact with the perlite surface and hence also with the boundary of the 10” pipe. This setup may have directly resulted in the formation of the void, whereas in the last three experiments, no pathway along the pipe wall of the experimental rig was seen and so this may be a more realistic scenario for releases into the bulk perlite. Voids along the wall were seen in Test 2 and especially Test 4 and 5. It is believed that these voids do not occur as soon as top valve is opened to let LNG into the chamber above the perlite. This is because:

- The measured pressure above and below the perlite layer was found to be similar throughout the experiment when LNG was released into the top chamber
- The thermocouples at the bottom of the perlite either did not show cryogenic temperatures, as in Test 2, or only showed cryogenic temperatures when the bottom valve was opened, Tests 1 and 3.
- The pressure traces above and below the perlite also matched for Test 3, where no obvious void was observed

Even though it is hypothesised that there was no immediate void formation when LNG was released into the top chamber, gas must have been able to permeate the perlite immediately. This is because the results clearly indicate that there was negligible pressure difference between the top and bottom layers as soon as top valve was opened. The results in Tests 6-8 indicate that the gas permeated along the inside wall of the 10” pipe, rather than through the bulk perlite, since a pressure difference was not measured in these later tests.

A clear pattern for the event(s) that triggered the void formation in Tests 1-5 is not obvious. The results do indicate that the formation of voids above the 5” orifice in Tests 4 and 5, probably did trigger the formation of voids higher up the perlite layer. If so, the relatively small loss of perlite below the layer in Test 3, was possibly insufficient to trigger void formation higher up. Another factor for Test 3, may be the relatively low pressures achieved throughout this test, e.g. ≤ 1.4 barg. While the pressure in Test 4 reached a maximum of 4 barg and consistently 3.2 barg when the bottom valve was opened. In Test 5, the final pressure was only 1.3 barg, but it did spike to almost 3 barg just prior to the valve being opened and was ~2 barg when the top valve was opened. However, there are other inconsistencies in the experimental data, that make it difficult to draw definite conclusions about the conditions that are pre-cursors to the void formation along the walls. For instance, in Tests 1-3, cryogenic temperatures were not recorded until the bottom valve was opened, whereas they were seen in Test 4, shortly after filling was stopped, while the base of the perlite in Test 5 cooled to cryogenic temperatures at some time between the end of filling and the opening of the bottom valve.

The results are clearer for why and when the void was formed during Tests 6,7 and 8. In these tests a clear void was formed outside the orifice in Tests 6 and 7, due to the momentum of the LNG that was released under pressure when the top valve was opened. The size of the void seems to be related to the overall flow rate, e.g. a bigger void was created in Test 7 through the 20 mm orifice, whereas the void was deeper in Test 6, when the exit velocity and flux (kg s^{-1} m^{-2}) of LNG though the 5 mm orifice was higher. It is also possible to explain the lack of an obvious void in Test 8, although there did seem to be a region of lower density perlite around the orifice when the perlite was removed. This is believed to be different, because the pressure increased significantly more slowly in this test, whereas the perlite instantly experienced a jet of LNG at 3.7 barg in Test 7 and a pressure of 5 barg in Test 6 as the top valve was opened.

The results in Test 6-8 also show that voids or cracks are not generated throughout the perlite layer as LNG comes into contact with warmer perlite and undergoes a rapid phase transition. Prior to these tests, it was conjectured that high pressure regions would form as the LNG was vaporised, which would propagate through any microcracks within the perlite and form an extended void. Even though the perlite was pre-cooled, the region at 150 mm depth had only cooled to a minimum temperature of -40°C in Test 6 and -10°C in Test 7. It is therefore likely that a backpressure was generated, that naturally restricted further flow of LNG, until an equilibrium was established. Consequently, any voids generated in the perlite from an orifice release will be self-limiting and certainly will not penetrate wider regions of perlite, such as the 300 mm used in these tests.

Gas permeation rates

An estimate of the gas permeation rate was calculated for each test by analysing the time that the liquid level reached each graduation mark in the 200 litre plastic vessel. The results for Tests 2,3, 4 and 5 are shown in Figure 9. This shows that the gas permeation rates for each of these tests is fairly uniform, although it did take about 10 s to reach a steady rate in Test 3. Note that gas was also collected in Test 1, but a smaller capacity plastic vessel used in the first test was filled in a few seconds.

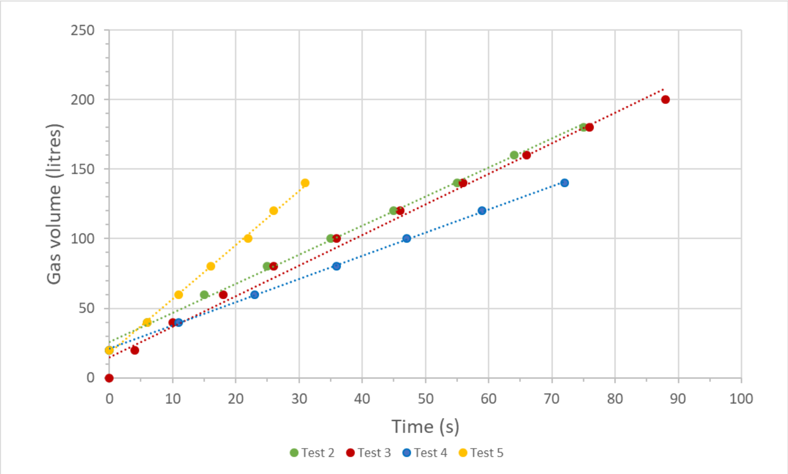

Figure 9: The volume of gas collected for Tests 2-5 as a function of time

The flow rates for Test 2 and 3 were calculated to be 2.1 litres s^{-1}. However, it is believed that these rates may be underestimated. In these two tests, the 10 mm pipework had an internal diameter of 8 mm and the collection vessel was situated 9.5 m from the experimental rig and so it is hypothesised that the flow was restricted. This theory is supported by flow rate calculations of this physical layout and the internal pressure (1.7 barg in Test 2 and 1.4 barg in Test 3) using Shell FRED. These calculations showed that a theoretical flow rate of 2.5 – 4 litres s^{-1} was possible for the given conditions. However, the pressure sensors on either side of the perlite layer for Test 2 and 3 also showed a pressure difference for approximately 100 s following the opening of the valve, which is not seen in the subsequent tests 4 and 5 after the pipe between the rig and the plastic collection vessel was replaced with a pipe with an inner diameter of 22 mm and a reduced length. Consequently, the permeation rate for Tests 2 and 3 is likely to be higher than the 2.1 litres s^{-1}

The flow rate for Test 4 was calculated to be 1.7 litres s^{-1} and therefore was probably less than that measured in Tests 2 and 3, if the flow in those tests was restricted. This is surprising considering the loss of perlite from the base and the apparent void seen in this test. In comparison, the flow rate during Test 5 was calculated to be 3.9 litres s^{-1}, significantly higher than Test 4, although the void formation appeared to be similar. Therefore, in these tests where the LNG was applied directly to the perlite, the gas permeation rate cannot be directly correlated to either the upstream pressure or whether there is a loss of perlite volume on the low pressure side. The permeation rate is likely to be dependent on the initial void formation in the perlite and hence how the perlite has been packed into the annulus space on a vacuum insulated tank.

The gas permeation results from the final two tests (7 and 8) are shown in **Figure 10**. In this graph there are three separate results for Test 8 that correspond to three separate periods when the bottom valve was opened. The highest flow rate was recorded during Test 7, when 3.2 litres s^{-1} were measured, whereas initially only 0.25 – 0.5 litres s^{-1} was recorded during Test 8 and no permeation was recorded during Test 6. Consequently, the gas permeation through a small orifice could be correlated to hole size as the orifice is increased from 5 mm to 20 mm.

In Test 8, the first two pressure cycles give a gas permeation rate of 0.25 - 0.5 litres s^{-1}. During the third cycle, gas permeation starts to occur at pressures closer to 1 barg; the initial gas permeation rate is 0.3 litres s^{-1}, but it increases to 1.7 litres s^{-1} over 100 s, without any increase in the top pressure. This appears to show that a permanent pathway for gas permeation has been created by the pressure cycling during the test. Overall, the second series of tests with a defined hole size shows more consistent behaviour when predicting gas permeation rates, although still with a large degree of variation. It is expected that the variable nature of how the perlite is packed (even in this relatively simple geometry) is the major factor on the variability in the results that were seen.

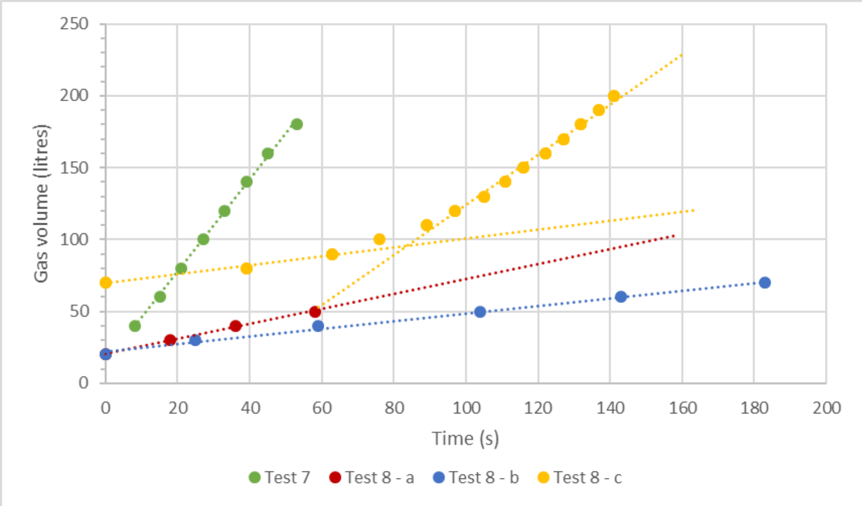

Figure 10: The volume of gas collected for Tests 7 and 8 as a function of time. Test 8 was split into three periods, corresponding when the bottom valve was opened

Permeation of LNG

One of the important criteria for updating the design criteria is whether actual liquid permeates the perlite. The results from Tests 1-5 indicate that voids could form adjacent to metal work, e.g. piping within the annulus, but these voids are unlikely to extend through a 300 mm layer of perlite. However, there is also evidence that cryogenic temperatures, approaching -160°C, were seen at the bottom surface of the perlite.

In some tests, such as Test 3 and Test 4, there is a sudden change, such as when the bottom valve is opened, which can be attributed to LNG passing to the bottom surface via voids. However, in many of the tests, there appears to be a more gradual reduction of temperature for the thermocouples located within the perlite. This was observed in the majority of the first five tests, where a temperature gradient of 0.2-0.3°C s^{-1} was measured. This rate of temperature decrease is too slow to reflect LNG travelling through voids, but equally is much quicker than general cooling if the LNG is just sitting on top of the perlite (calculated to be 0.045 °C s^{-1} for a thermal conductivity of 0.04 W m^{-1} K^{-1}). This result seems to show LNG slowly permeating (and vaporising) as it descends through the perlite to provide the additional cooling demonstrated by these results.

It also appears that the rate of cooling through the perlite is enhanced when the pressure above the top surface of perlite is raised. This can be seen by plotting the time that each thermocouple reaches -60°C (generally corresponding to the largest rate of change of temperature) is plotted as a function of depth. The results in Figure 11 show and compare the results for Tests 2 and 3 (at approximately 1 barg) and Test 1 and 5 (at approximately 2 barg). Note that the results for Test 4 is not shown, because the thermocouple responses were unstable after filling stopped. The analysis shows that the time for the perlite at a depth of 200 mm to reach -60°C is much quicker for the higher pressure. The graph also shows a very gradient for the two pressures between 200 mm and 280 mm for 2 barg and 100 mm and 200 mm for 1 barg, which can be correlated to a reduction in the pressure in Tests 1 and 5 after approximately 1000 s. The result for 280 mm is plotted for Tests 2 and 3, although these times are reduced because of a sudden change in temperature, probably due to flow through voids, as commented at the start of this section.

It was more difficult to ascertain if any liquid permeated through to the bottom surface of the perlite in the last three tests. This is not surprising considering the surface area in direct contact with the LNG was significantly less, even after the generation of voids around the orifice. As reported previously, no gas permeation was reported in Test 6 and the thermocouple at 250 mm depth only reached -30°C after 4 hours. In Test 7, the thermocouple at 250 mm does show cooling to -30°C over a relatively short period (approximately 500 s) when compared with Test 6. However, the experiment was stopped at this time, because the gas collection container had been filled and so no conclusion about liquid permeation can be made. The result for Test 8 is slightly different; gas permeation was measured after the initial pressure cycle, but the response at 250 mm does show a more similar trend to that seen in the first five tests. Specifically, the temperature at the base of the perlite was seen to decrease from -40°C to -140°C within 1000 s. In addition, the time required for thermocouples at depths of 150 mm and 250 mm within the perlite (T2 and T5) to reduce by -60°C is 1500 s, which is faster rate of change than that shown in Figure 11. This is perhaps not surprising considering the pressure exceeded 5 barg, even if it was not for the whole duration of this time period.

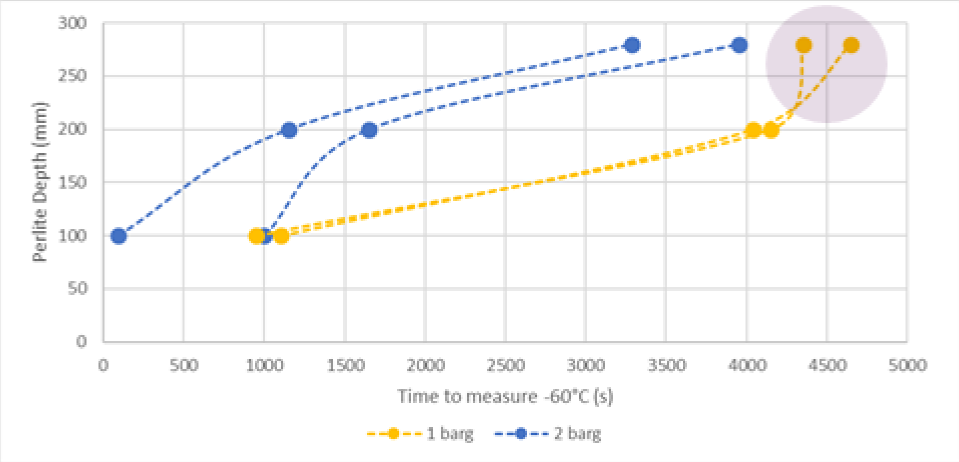

Figure 11: The time for perlite to reach -60°C as a function of pressure for Tests 1-3 & 5. The highlighted region shows when voids may have formed and allowed the thermocouples at deeper layers to cool more quickly

Overall, it is not clear if liquid can permeate through the perlite if voids do not form, however the results do show that cryogenic temperatures can propagate through the perlite faster than would be expected by normal conduction if sufficient LNG volume is in contact with the surface of the perlite. The results in Figure 11 suggest that the temperature gradient is 110-135 mm hour^{-1} during the application of LNG at 1 barg. This implies that the outer surface of a vacuum insulated tank with 300 mm of perlite will reach LNG temperatures after 2 to 3 hours following a loss of containment at the inner surface. The results indicate that this would be quicker at higher pressures and significantly quicker, of course, if a void was formed at the edge of internal pipework, that allowed the LNG to flow under gravity to the outer tank skin.

Application to real tanks

First, it is clear that internal pipework within the annulus space will have a large influence on the permeation of both gas and liquid, especially if this allows the LNG to fall under gravity alongside the metal. In this case, the results from Tests 1-5 show that the pressure on the outer surface of perlite will match the inner pressure and hence cause a loss of gas through the vacuum plate. Even in the more realistic experimental design using 5-20 mm orifice, gas permeation was seen for the 20 mm, and eventually for the 10 mm orifices. Therefore, it is reasonable to assume that any failure at the inner surface with an orifice of approximately 10 mm or greater will result in gas permeation and hence loss of gas through the vacuum plate. In these cases, the maximum rate of gas flow was only measured to be 2 – 3 litres s^{-1} and so a large orifice for the vacuum plate, e.g. 6” is probably not required, even if the inner failure was from a 50 mm orifice.

Gas permeation to the vacuum plate is predicted to occur even in long horizontal tanks, where the vacuum plate may be as much as 50 m from the leak source, because the perlite directly adjacent to the tank walls is expected to have a reduced density allowing the gas to permeate more easily. However, the density of perlite in this region should still be sufficient to restrict the flow of liquid, especially without the influence of gravity. Consequently, any LNG that permeates to the outer jacket is unlikely to move laterally along the inner surface of the outer jacket wall.

The tests with a 5” orifice (Tests 3 – 5) showed evidence that perlite would be lost through a vacuum plate. However, if the perlite is packed to the correct density in the vicinity of the plate, then there is sufficient friction to ensure that the amount of lost perlite will be minimised. Consequently, the free flow of perlite through the vacuum plate is not expected to happen.

The results did show that a void was created around the exit plane of both a 5 and 20 mm orifice, but only if the perlite was suddenly exposed to LNG at pressure. However, this void did not extend a significant distance into the perlite. This means that there was no evidence of any rapid phase changes of LNG when “warmer” perlite was exposed at the edges of the void. Consequently, the release of cryogenic liquid will not propagate through the whole perlite layer, through the “explosive” creation of a leak path. In addition, the void appears to require a starting pressure, for example, no obvious void was seen in Test 7. This could mean that if a failure started as a pin-hole, it is possible that only a small void would be generated initially, but this void might not grow if the orifice grew larger (because the perlite would not be instantly exposed to a higher pressure if failure become worse).

One of the key requirements in the design of vacuum insulated tanks, is whether the outer tank could be exposed to cryogenic temperatures and hence would require the selection of stainless steel at additional cost. The experimental results appeared to show that LNG did not permeate through 300 mm of perlite, but cryogenic temperatures were seen to propagate through the perlite at speeds much greater than would be expected purely by thermal conduction. It is estimated that it would take 2-3 hours for cryogenic temperatures to travel across a 300 mm layer of perlite, but this time span could be significantly shorter if the LNG was supplied to the perlite surface at pressures greater than 1 barg or a void was formed adjacent to pipework. This effect was not seen when using a 5 mm orifice in Test 6.

Unfortunately, it was not possible to repeat these tests with a reduced perlite thickness to be able to interpolate the effects for different tank designs. However, it is believed that the majority of effects and conclusions outlined above would still be valid if the perlite was greater than 150-200 mm (half the 300 mm chosen in these tests). The one exception may be how much perlite is lost through the vacuum plate. In Test 4 and 5, the perlite lost from the bottom surface was as much as 100 mm deep. If the overall perlite thickness was only 150 mm, then it is conjectured that the whole perlite layer in the vicinity of the vacuum plate may be lost.

Conclusions

This report summarises a series of medium scale experiments that were commissioned to address uncertainties about the permeation of LNG gas and liquid through perlite insulation. A better understanding of the permeation would allow a more informed decision to be made suitable mitigation in the current design, such as the use of stainless steel rather than carbon steel for the outer tank wall. Overall the following conclusions can be made:

- The released LNG created voids along the boundary between the steel wall of the test rig and the packed perlite in several of the tests. These voids resulted in sudden changes in temperature and permeation of gas (and potentially liquid) to the downstream side of the perlite. Therefore, pipework within the annulus space may directly affect LNG permeation; even allowing LNG to travel directly to the outer jacket. To minimise the flow of LNG in the event of a failure, it is suggested that the amount of pipework is reduced, especially in the lower sections of the tank, and where possible, it should be angled to reduce the effect of gravity.
- A sudden release of pressurised LNG into bulk perlite will generate a void with a volume related to the flow rate and exit velocity of the release. However, the exposure of "warmer" perlite at the edges of the void did not cause further gas generation and hence expansion of the void. Consequently, the release of cryogenic liquid will not propagate through bulk perlite via a series of small rapid phase transitions.
- The experiments demonstrated that some perlite was lost from a 5" diameter orifice designed to simulate a vacuum plate opening. This could result in reduced perlite thickness in the immediate vicinity of the vacuum plate, however, the perlite is not expected to flow freely through the orifice due to the density at which it is packed.
- The highest measured gas flow rate was 3.1 litres s^{-1} (20 mm orifice). For smaller orifices, there was either no gas permeation or gas permeation was only seen after repeatedly cycling the pressure above 5 barg. The size of the vacuum plate for double skinned tanks should be re-evaluated based upon maximum design pressure and for the largest credible leak scenario.
- The results suggest negligible gas permeation for a failure with a hole size of 5 mm. In addition, the minimum temperature experienced at a depth of 250 mm for this hole size was no lower than -40°C after 5 hours.
- Cryogenic temperatures were seen to propagate through the bulk perlite. Although this is not believed to be due to liquid permeation, the speed was much greater than would be expected by only thermal conduction. It is estimated that a 300 mm layer of perlite would reach -160°C after 2-3 hours when the leak size was 10 mm and above. The design strength of the outer jacket should be checked for exposure to temperatures below -60°C, if a 10 mm hole size is considered to be a credible hole size.

The conclusions above should be valid for tank designs with a perlite thickness of 200 mm or above. Although, as a depth of 100 mm of perlite was lost above the 5" orifice designed to simulate the vacuum plate in two experiments, then it is conjectured that the whole perlite layer in the vicinity of the vacuum plate may be lost in this case. This effect can be minimised by reducing the size of the vacuum plate.

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