WATERPROOF INSULATION MATERIALS

R.Z.J. Lang, J. Moorhouse and G.J. Paul.*

Insulation applied as a lining to a bund around a cryogenic fuel store offers a means of reducing vapour evolution following a spill of liquid. This paper examines the water take up by insulating concretes treated in a variety of ways, and the influence that water content has on boil-off from these materials.

INTRODUCTION

In any application where thermally insulating materials are employed it is essential that they maintain their insulating properties under the conditions of use. This can be difficult when insulation materials are directly exposed to outdoor conditions, particularly if the materials are not waterproof. The presence of water not only reduces the insulating properties, but can also lead to spalling under frost conditions if water contents are high.

Insulation of impounding areas around liquid gas storage tanks is a typical outdoor application. The purpose of the insulation in this case is to reduce the evolution of flammable vapour following a spill of fuel and hence reduce the spread of the vapour cloud and the chance of a fire occurring. A suitable insulation material for such situations must maintain good insulation properties over a reasonable life as well as being mechanically stable, capable of sustaining light traffic on its surface, and fire retardant. It should also be relatively inexpensive and easily applied.

Lightweight concretes meet these requirements and they can be cast in situ or sprayed onto surfaces. However, their low densities (typically 0.6 - 1.6 g/cm^3), which can be achieved either by foaming or by the use of lightweight aggregates, result in a porous material. If steps are not taken to seal such materials in some way then under wet conditions they will take up water.

*British Gas Corporation, Midlands Research Station, Solihull.

WATERPROOFING TECHNIQUES

Techniques for waterproofing lightweight concrete materials have been examined in a recent paper by McClune et al (1). The general findings were that for samples immersed in water, water take-up at equilibrium was approximately related to the material density for a range of different lightweight concretes. All lightweight concrete samples absorbed in excess of 14% by volume. Two different approaches were attempted to reduce the water take up, i.e. surface sealants and integral waterproofers.

The effectiveness of a number of surface sealants when applied to lightweight concrete samples was examined. The conclusion reached was that surface sealants are only effective if a perfect coating is achieved. Any small imperfections allow water ingress and the subsequent drying is extremely slow due to the sealing layer. In practice it is unlikely that a perfect coating could be achieved and moreover thin layers are vulnerable to mechanical damage. This could be overcome by application of much thicker coatings but this has a deleterious effect on the insulation properties at the surface, and is more costly.

The effectiveness of integral waterproofers was examined using various agents incorporated into lightweight concrete mixes. Commercial waterproofing additives were generally found to be ineffective for use in lightweight materials. Two techniques were found to be of some value and these were silicone impregnation and use of a latex additive. Silicone impregnation of aggregate or the cast material reduced the rate of water take up, but in time the silicone was leached off the material. This resulted in a similar equilibrium water content to those which were untreated. Latex added to the water when preparing samples was found to significantly reduce the rate of water take up, particularly when used to replace most of the gauging water.

Two techniques were therefore found which showed a reasonable degree of waterproofing. All of the tests were however carried out by subjecting samples to total water immersion. Whilst this is a realistic test for insulation which may occasionally be subject to standing water, for example on the floor of a bund, it is undoubtedly severe for insulation on a vertical surface. Continuous exposure of samples to outdoor weather conditions is a more meaningful test for such applications.

Selected samples, prepared in a similar manner to those described elsewhere (1) have therefore been subjected to outdoor exposure tests. The results of these tests are shown in Figures 1 and 2. Details of the test samples are given in Table 1. Large variations in water content occur for untreated lightweight concretes due to changes in weather conditions; peak values of 23% water by volume were found. Silicone treatment of a sample, achieved by a 5 minute total immersion, restricted the water content to less than 10% by volume. Addition of a latex (i.e. styrene-butadiene rubber) solution to the gauging water of the mix can be seen to result in a take up of less water than an untreated sample, the improvement increasing with increasing quantity of rubber solution. Maximum water take up levels can be restricted to around 10% by replacing 75% of the gauging water with latex solution. This is consistent with the previous work under total water immersion conditions (1).

These techniques therefore may give a satisfactory measure of waterproofing when the material is not subjected to standing water, but this depends on the water content which is acceptable, whilst still maintaining satisfactory insulation properties.

TABLE 1 — Details of Samples used in Outdoor Exposure Tests

Sample designation	Treatment	nominal size	dry density g/cm^3
C1	Control sample. Untreated lightweight concrete.	7.5cm cube	0.86
C2	Control sample. Untreated lightweight concrete.	7.5cm cube	0.82
S1	Silicone treated lightweight concrete sample immersed in silicone agent for 5 minutes prior to test.	10cm cube	1.15
S2	Silicone treated lightweight concrete sample immersed in silicone agent for 5 minutes prior to test.	10cm cube	1.15
L1	Lightweight concrete made with latex solution replacing 50% of gauging water.	7.5 cube	0.87
L2	Lightweight concrete made with latex solution replacing 50% of gauging water.	7.5 cube	0.80
L3	Lightweight concrete made with latex solution replacing 75% of gauging water.	7.5 cube	0.85
L4	Lightweight concrete made with latex solution replacing 75% of gauging water.	7.5 cube	0.80

INSULATION PROPERTIES

If a spill of a cryogenic liquid, e.g. LNG, occurs onto an insulation material, heat is removed from the material causing boil-off of vapour. As the cold penetrates into the material the heat flow subsides. The heat transfer process is therefore time dependent, and for semi infinite materials with temperature independent thermal properties it is described by the following relationship (2).

$$Q = \frac{kT}{\sqrt{\alpha t \pi}}$$

$$= B\, t^{-1/2}$$

It is convenient to compare insulation properties by carrying out laboratory scale spills of cryogens onto small samples of materials. This has been done for a variety of materials under dry conditions (3), but information on boil-off from moist materials is limited to that from soils and sand.

Data for sand reported by Drake et al (4) show a reduction in boil-off rates with increase in moisture content from B = 270, dry, to B = 240 with 3% moisture.

This trend is confirmed for soils (4) where an increase in moisture content from 1.9 to 7.5% caused a decrease in B from 260 to 157. For a 'very wet soil' however Humbert - Bassett et al (5) observed a significant increase in boil off rates, from B = 530, (dry soil), to B = 757, (very wet soil).

In highly porous materials such as soils, it is likely that LNG penetrates into the material thus increasing the effective surface area. Small additions of water to a dry soil will probably ensure that when soil freezing occurs this penetration will be reduced, and hence boil-off rates will be reduced. However when water contents are high, the substantial increase in thermal conductivity of the wet material will serve to generate high rates of boil-off.

This explanation has been put forward in published literature to explain the experimentally observed influence of soil water content on boil-off rates.

In view of the lower porosity of insulating concretes compared to that of soils, it is to be expected that the published data for soils will not present a good indication of the boil-off rates from wet insulating concretes. An experimental study has therefore been made of boil-off rates from insulating materials having different water contents. Such information can be used to determine an acceptable water content from a boil-off standpoint and thus determine whether the waterproofing techniques can meet the acceptability criterion.

EXPERIMENTAL TECHNIQUE

An apparatus was devised to provide boil-off information for cryogenic liquids spilled instantaneously onto samples of insulating materials, having a range of water contents. The main requirements for the experimental technique were that it should be able to execute rapid spills of known quantities of cryogen, and provide accurate and reproducible boil-off data during each experiment.

The apparatus is shown schematically in Fig. 3. Test samples were cast as thin discs (22.9 cm diameter and 2.5 to 5 cm thick) and set into polystyrene tubes, which provided containment for the spilled liquids, without contributing significantly to the level of boil-off (less than 5%).

Test samples were placed on the pan of an Oertling Metronic electronic top-pan balance, having a maximum capacity of 12 kg with a resolution of 1 g, and analogue output proportional to the weight on the pan. A wooden cradle was mounted on top of the polystyrene walls of each container, and this supported a glass dewar. This dewar was open at the bottom and, in order to restrain the quantity of cryogen to be spilled, a thin plastic membrane was attached. Spills were actuated by rupturing the membrane from above using a sharp blade.

At the outset of each test the liquid cryogen, liquefied natural gas (LNG), was transferred to the dewar. The quantity to be spilled was measured by the balance. Efforts were made to ensure that the composition of the LNG used in the tests was similar and in fact the average composition was 90% Methane, 8% Ethane and 1% Propane by volume in the vaporised liquid. Rupture of the membrane produced a rapid spill, and boil-off rates were determined for very early times from the instant of the spill, (usually from 1 second onwards). Mass fluxes ($kg/m^2 s$) were determined at specified time points from continuous weight measurements, recorded using a computer-controlled data acquisition system.

Experiments were carried out on both lightweight and ordinary concrete samples. These were carried out when the samples were in a laboratory dry condition, when they were saturated with water, and also for lightweight concretes at intermediate water contents. The saturated condition was obtained by soaking the test samples in water for several weeks, and the intermediate water contents by controlled drying of the saturated samples.

The drying down of the samples was carried out in two different ways. One technique was to allow the sample to dry out from the top surface only, which represents what would happen in an outdoor application. The other technique was to allow a sample to dry down to a chosen level and then confine it within a sealed plastic bag for a minimum of 24 hours in order to encourage a more uniform water distribution throughout the sample.

RESULTS OF EXPERIMENTS

Details of the concrete samples that were tested are given in Table 2, with a list of experiments that were carried out in Table 3. A typical example of a mass time curve for a test on wet lightweight concrete is shown in Figure 4. It illustrates the steady mass prior to the spill, the slight rise in mass as the membrane was ruptured due to the action of the blade used to achieve the rupture, and the mass decrease during the boil-off process. In some tests, immediately after the spill, some LNG splashed out from the polystyrene cylinder. Hence not all the initial mass loss is actually due to the boiling process. Data such as that in Figure 4 were used to derive boil-off rate data as shown in Figure 5. Since the boil-off rate is proportional to the gradient of the mass/time curve, small inaccuracies in mass are magnified. From similar boil-off rate data for each of the tests, lines have been drawn which represent the data points and these are shown in Figure 6 for dry materials, Figures 7 and 8 for wet lightweight concretes, and Figure 9 for wet ordinary concrete.

The potential value of a dry lightweight concrete as an insulator compared to a dry ordinary concrete is clearly shown. The influence of water content on boil-off rates is very significant for both lightweight and ordinary concretes. Other points are also evident from the data obtained. For the water saturated samples a peak in boil-off rate occurs at around 0.35 to 0.38 $kg/m^2 s$. Further the gradients of the boil-off time curves show a steeper slope than the $t^{-1/2}$ dependency observed for dry materials.

Because of the differences in gradient and the occurrence of peak boiling fluxes it is not easy to make a simple comparison between samples from individual tests. The most convenient way is to determine the average boil-off rate over the initial part of the spill, since this is generally of most interest. In determining this average the first 3 seconds have been ignored in order to remove the possible influence of splash losses on the figures. Average boil-off rates determined in this way are shown in Table 3, and graphically in Figure 10.

Because of the limitations of the data logging process which cannot resolve intervals of less than 1 second, time zero could be in error by $\pm \frac{1}{2}$ second. This represents an uncertainty in the data which can lead to an error in average boil-off rate of up to 3%. Ordinary concrete shows almost a two-fold increase in boil-off rate between air dry and saturated, whilst lightweight concretes increase by a factor of about 4 over the same range. The differences between the lightweight concrete samples dried in different ways is small.

TABLE 2 — Details of Test Samples used in LNG Boil-off Experiments

Sample Material and Designation	Mix Design	Size	Dry Density g/cm^3
Lightweight concrete LW1	3:3:3:3:4 by volume Medium Aglite: Fine Aglite: Micafil: Mixed Perlite: Portland Cement	9" diameter disc set in polystyrene cylinder	1.16
Lightweight concrete LW2	"	"	1.17
Standard concrete SC1	5:7:12 by volume Cement: Sand: Gravel	"	2.24
Resin concrete	2:2:3 by volume Medium Leca: Fine Leca: Fire retardant polyester resin	"	1.22

The overall influence of inherent water content on boil-off rates of cryogenic liquids is therefore large. It is however encouraging to observe for lightweight materials that if the water content could be maintained below about 10% then boil-off rates would only be increased by about 50% above the air dried condition and this is still significantly better than for an air dried ordinary concrete.

RESIN CONCRETES

Two techniques have therefore been found to achieve a substantial degree of waterproofing. It was however recognised that an alternative approach would be needed if any further improvement was to be achieved. One alternative solution is to use lightweight aggregates as before, but utilise resin as the binding material instead of cement. In this way the low permeability of the resin is employed to restrict water ingress. Investigations into the feasibility of such an approach have been confined to 'resin concretes' made from fire retardant polyester resins and an expanded clay aggregate; this material is available in a range of size gradings (fine, medium and coarse). Final densities of between 1.0 - 1.4 g/cm^3 have been achieved with these materials.

In order to assess the qualities of these materials, samples of resin concrete have been subjected to water immersion tests and boil-off measurements as described above. Typical results for water take-up (8 cm cube samples) are given in Figure 11, where they are compared with other materials. It can be seen that the rates of water take up are very much lower than for previously considered materials, and a much lower equilibrium water content is indicated.

TABLE 3 – Details of Boil-off Experiments on wet Materials

Test Sample	Water Content of Test Sample % by volume	Average Boil-off Rate between 3 and 30 seconds $g/m^2 s$
LW1	21.9 (saturated)	128
LW2	23.0 (saturated)	138
SC1	10.5 (saturated)	208
LW1	19.4	108
LW2	20.6*	110
LW1	15.3	78
LW2	17.1*	78
LW1	5.9	39
LW2	7.8*	38

* Samples placed in a sealed plastic bag for a minimum of 24 hours before boil-off test to encourage a uniform water content.

NB Several tests were carried out on each test sample in an air dry condition.

Since water take up is very small, boil-off measurements were only made on dry samples. The results of these experiments are presented in Figure 12 for spills of LNG where they are compared with results for standard concrete and a typical lightweight concrete. The performance of resin concrete in the boil-off tests can be seen to be slightly better than that of air dry lightweight concretes.

In order to be suitable for use as an insulation lining of a bund, three other properties are important. These are the mechanical strength of the material, the bond strength between the insulation layer and the substrate material, and the resistance to cold shock conditions. Resin concretes have therefore been examined for these three properties.

In bond strength tests cores of bonded material, with metal end pieces glued into place, have been subjected to tensile loads of up to 4480 KN/m^2. Six samples were tested and the resin concrete/standard concrete bond remained intact in all cases. In three tests the standard concrete part of the core failed at 2760, 3100 and 3450 KN/m^2, and in the remaining tests failed through a bond holding the metal end pieces at forces of 1380, 2760 and 4480 KN/m^2. Although these tests did not establish the strength of the bond in question, they have shown that it is very strong, at least as strong as the standard concrete substrate.

Results of 28 day compressive strength tests have been reported (1) on a range of insulating concretes. Resin based materials have a significantly higher compressive strength than cement based materials of a similar density. For a polyester resin the increase is by a factor of 2 whilst for an epoxy resin the increase is by a factor of 3.5.

In order to assess the possibility of a resin concrete coating being affected by thermal shock, a sample bonded to a standard concrete substrate was covered by liquid nitrogen for a 30 minute period. On inspection the sample exhibited a few hairline cracks, but nothing more serious. Those samples used in boil-off tests showed similar behaviour. It is evident therefore that resin concretes will maintain an adequate bond strength during a cold shock and will not suffer from surface spalling.

CONCLUSIONS

The intention of insulation materials is to reduce vapour evolution rates in the event of a spill. It is therefore highly desirable that materials for use outdoors are capable of maintaining their insulation properties in all conditions. In order to do this a high degree of waterproofing is necessary, but the degree required will depend upon their conditions of use.

Cement based lightweight concretes are vulnerable to water take-up under wet conditions, but for vertical surfaces which are not subject to standing water conditions two techniques have been shown to be potentially useful. Silicone treatment can maintain water contents at low levels, but it is likely to require retreatment at regular intervals. Only longer term testing will prove the necessity for this. Addition of a latex solution to a cement based mix has also proved to be a potentially valuable technique, maintaining similar low water content levels.

For outdoor conditions where standing water may occasionally be present and hence a higher standard of water proofing is required, then resin based concretes are the most suitable type of material. They offer a wide flexibility in the choice of aggregate since it is the low permeability of the resin which imparts its water resistant properties. High mechanical strength and bond strength are also possible with this material. These particular concretes also show a reasonable resistance to cold shock.

For an insulation material to be viable it must be relatively inexpensive, and convenient low-cost techniques for application should be available. Lightweight concrete layers can be sprayed onto existing concrete substrates and these materials are cheap; the layer thickness therefore is not critical, and may be determined by the method of application.

Resins are costly, and it is desirable to manufacture resin concrete mixes which are as rich in aggregate as possible, consistent with a means of application and good adherence to the substrates. In situ casting is feasible for horizontal surfaces, whilst for vertical surfaces spray application is desirable. Trials with various mixes of resins and aggregate have indicated that spray application is possible with some types of aggregate, and this is an area that requires further larger scale work.

Since resin concretes are costly, it is fortunate that thinner layers are attainable than those produced by spraying lightweight concretes. Thinner layers can be tolerated since it is in the initial period of contact that boil-off rates are highest, and this is when the cold has penetrated only a short distance into the insulating material.

Estimates have been made of boil-off rates associated with layers of different thicknesses of a typical resin concrete applied to an ordinary concrete substrate. These have been done using the calculation technique described elsewhere(3) using estimated values for the thermal properties of the two materials. The results which are shown in Figure 13 illustrate the relative value of different coating thicknesses. This calculation technique can be used to optimise the balance between costs and the required level of safety in any proposed application.

SYMBOLS USED

$B = kT/\sqrt{\alpha\pi}$

k = Thermal conductivity (kW/mK)

Q = Heat flow ($kW/m^2 s$)

T = Initial temperature difference between cryogenic fuel and insulation (K)

t = Time from spill (s)

α = Thermal diffusivity (m^2/s)

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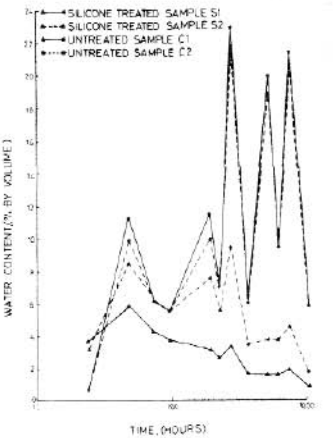


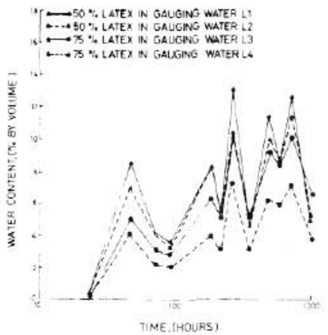
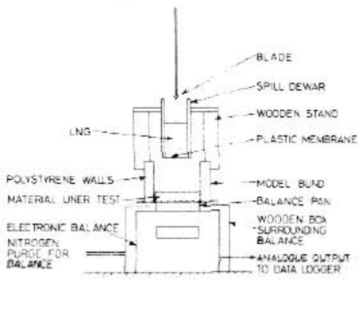


Figure 1. Outdoor exposure of silicone treated lightweight concrete

Figure 2. Outdoor exposure of lightweight concrete containing latex



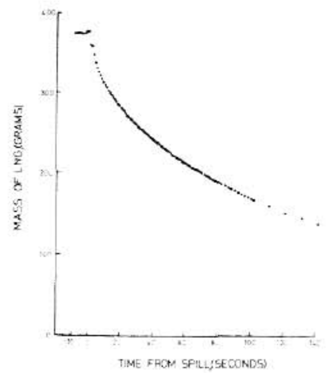


Figure 3. The experimental system

Figure 4. Typical mass/time curve (Wet lightweight concrete)

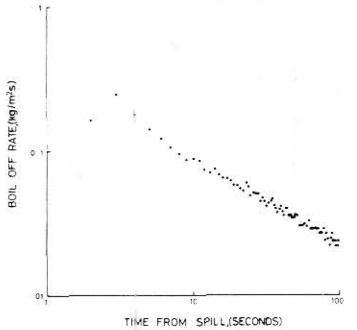


Figure 5. Typical boil-off rate data (Wet lightweight concrete)

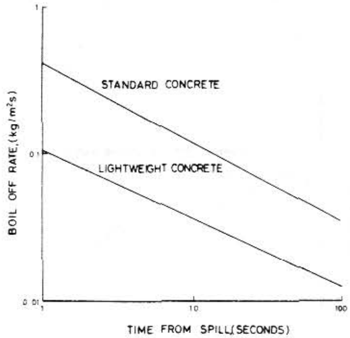


Figure 6. Boil-off rates from dry materials

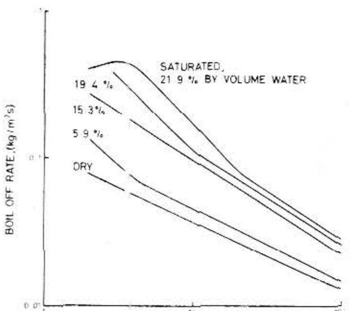


Figure 7. Influence of water content on boil-off rate for sample LW1

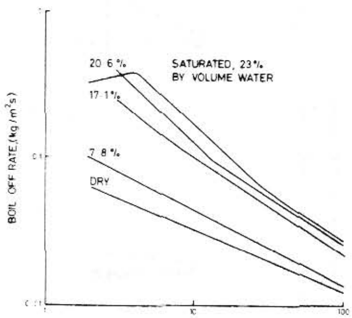


Figure 8. Influence of water content on boil-off rate for sample LW2

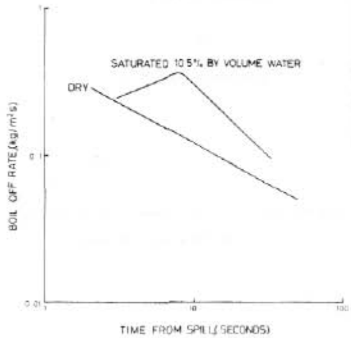


Figure 9. Boil-off from saturated and dry standard concrete

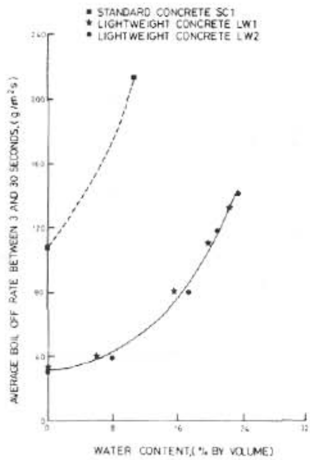


Figure 10. Effect of water content on average boil-off rates

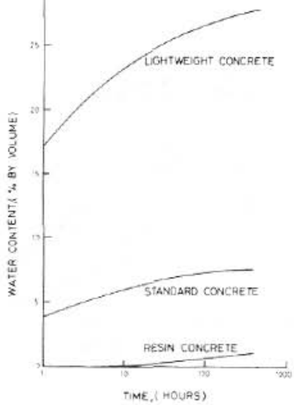


Figure 11. Water immersion tests on different materials

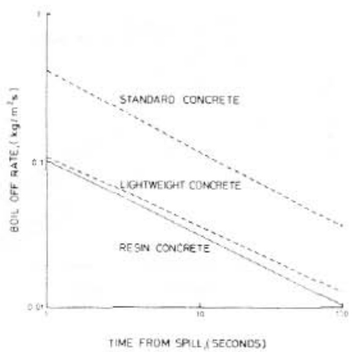


Figure 12. LNG boil-off rates from resin concrete

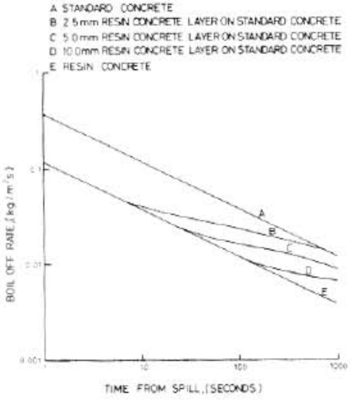


Figure 13. Calculated boil-off rates from composite materials