

Damage quantification and durability assessment of concrete at cryogenic temperatures

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Abstract

This study seeks to quantify the damage of concrete of different mix designs subjected to cryogenic temperatures, using novel non-destructive techniques. The techniques include x-ray computing tomography (XRCT), nuclear magnetic resonance (NMR) and scanning electron microscopy (SEM). Furthermore, the durability of the concrete mixes was assessed using transport properties such as water permeability and chloride permeability. The study is aimed at investigating design methodologies that improves damage resistance of concrete under cryogenic conditions, with a view to utilizing concrete for primary containment of liquefied natural gas (LNG). This would lead to huge cost savings compared to 9% Ni steel, which is currently used. Concrete cores, 2.5 cm diameter by 5 cm long, were used for the microstructural tests, while 150 mm concrete cubes were used for the permeability tests. These were produced using different mix designs involving river sand siliceous or manufactured limestone sand as fine aggregates. While limestone, sandstone, trap rock and light weight aggregate were individually used as

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coarse aggregates in the mixes. The samples were cured under water for 28 days and thereafter frozen to cryogenic temperature (-165°C) in a temperature chamber. Damage quantification involved assessment of microcracking and 3-D distribution of damage in cryogenic concrete by evaluating crack density and crack widths from SEM and XRCT images. Moreover, the NMR relaxation of hydrogen nuclei of pore water indicated the pore-size distribution. Studies are in progress to provide an improved understanding of how changes in the microstructure of cryogenic concrete affect its behaviour at the larger scale.

Keywords: Chloride permeability, microcracking, scanning electron microscopy, water permeability.

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Background Information

- Investigating concrete behavior at cryogenic temperatures ($\leq -165^{\circ}\text{C}$).
- ✓ Utilizing concrete for direct containment of liquefied natural gas (LNG).
- ✓ Traditional LNG tanks utilizes 9% nickel steel walls and floor for the inner containment tank – becoming increasingly expensive.
- ✓ Hence, this research seeks to achieve safe and economical storage of LNG.
- Literature review ^{1,2} shows that concrete properties generally improve at cryogenic temperatures (depends on moisture content & mix design).
- Research is in progress to Investigate concrete mixture design(s) that resists damage during cryogenic freezing.

Research Objectives

- This work seeks to provide an improved understanding of concrete behavior at cryogenic temperatures as there is insufficient information in this area. The specific objectives of the research are:
 - ❑ To improve our understanding on design methodologies to produce concrete that resists cracking induced by thermal stresses during cryogenic freezing.
 - ❑ To quantify the damage of concrete of different mix designs subjected to cryogenic temperatures using novel non-destructive techniques such as x-ray computing tomography (XRCT), nuclear magnetic resonance (NMR) and SEM.
 - ❑ To improve our understanding of how changes in the microstructure of cryogenic concrete affects its durability and behavior at the larger scale.

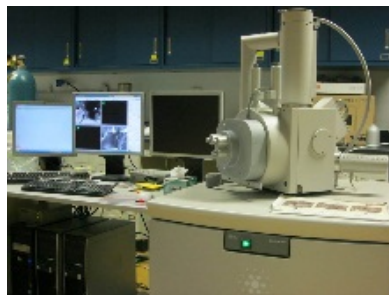
Research Methodology

- Concrete made from different mix designs are utilized for experiments.
- ✓ Current phase involves limestone, sandstone, trap rock and light weight aggregate as coarse aggregates and river sand siliceous as fine aggregate.
- ✓ The coefficient of thermal expansion (CTE) of the aggregates is in the range of 5 to 12 x 10⁻⁶ °C⁻¹ compared to 18 to 20 x 10⁻⁶ °C⁻¹ for saturated cement paste.
- ❑ *The differential CTE of concrete components leads to the potential for deleterious CTE mismatch induced stresses at cryogenic temperatures.*
- ✓ Considered in our quest for damage-resistant concrete mixture design (Obj. 1).
- Initial experiments entail cooling concrete samples from room temperature (20°C) to cryogenic temperature (-165°C) using a cooling rate of 3°C per minute.
- ✓ Photo on next slide shows concrete samples in the temperature chamber used.



Concrete samples in LN₂-cooled temperature chamber

- ✓ Concrete sample sizes are chosen to suit machine/equipment employed for post-freezing tests. NDTs employ concrete cores of 2.5 cm diameter by 5 cm long, in contrast to 150 x 150 mm cubes used for durability (permeability) tests.
- ✓ After cryogenic freezing, some frozen concrete samples are stored in corafoam insulation material to prevent temperature changes – e.g. before SEM imaging.
- ✓ The afore-mentioned cooling rate employed in the temperature chamber is in contrast to 0.017°C/min (1°C/hr – 8 days) typically used for LNG tanks.
- ✓ Higher cooling rate employed to encourage micro-cracking – for mix screening!
- ✓ The damage potential would also be evaluated as a function of temperature, cooling rate, and more concrete mixture designs in subsequent experiments.
- Damage quantification involves assessment of micro-cracking & 3-D distribution of damage from SEM & XRCT images.



SEM equipment



XRCT equipment

- ✓ Evaluate crack density & crack widths using advanced image analysis software.
- ☐ It also involves evaluating changes in pore size distribution using NMR relaxation of hydrogen nuclei of pore water (Obj. 2).

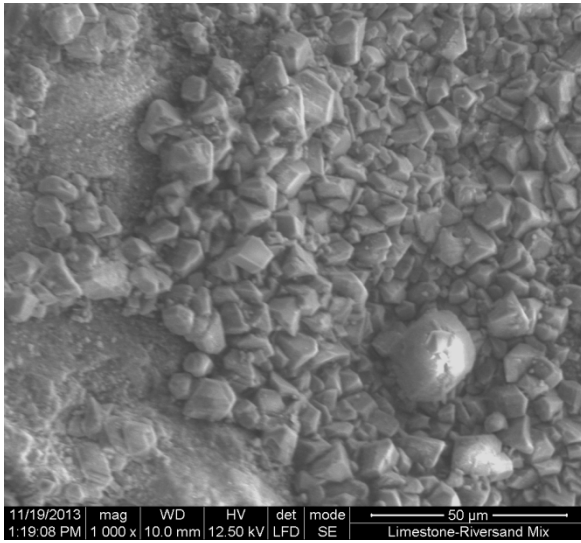


NMR equipment

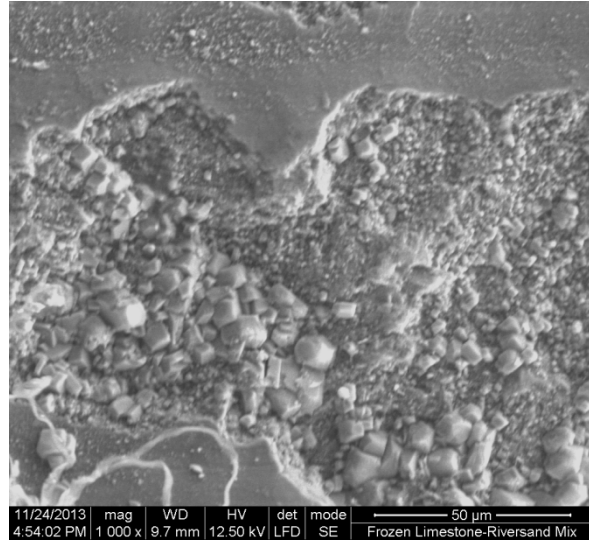
- The durability of the concrete mixes is assessed using transport properties such as water & chloride permeability (Obj. 3).
- ✓ Water permeability tests were conducted using depth of penetration method (BS EN 12390-8)³ on thawed cubes as opposed to use of flexible wall permeameters as in some previous studies⁴⁻⁶ due to the low permeabilities involved.
- ☐ Durability is linked with the state of micro-cracking. Hence, damage observed in NDT tests is validated with results of water and chloride permeability tests.

Selected Results – SEM Data

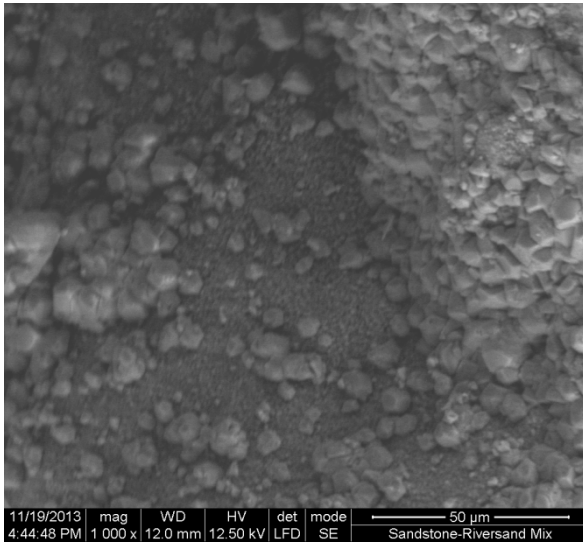
- In view of the heterogeneity of concrete, several different areas were scanned, with the images shown herein deemed representative.
- There is no clear visible evidence of microcracking in the concrete mixtures from the SEM images.
- ✓ One can anticipate this, as one-time freezing may not be sufficient to cause significant damage apparent in microstructural observation, as opposed to freeze-thaw cycling.
- ✓ Moreover, the SEM technique was employed for studying and comparing the surface morphologies of the mixes using specified marked points, which poses further difficulty in the imaging of microcracks developed because of one-time freezing since the specific points chosen in advance of the freezing process might not be the location of any damage.



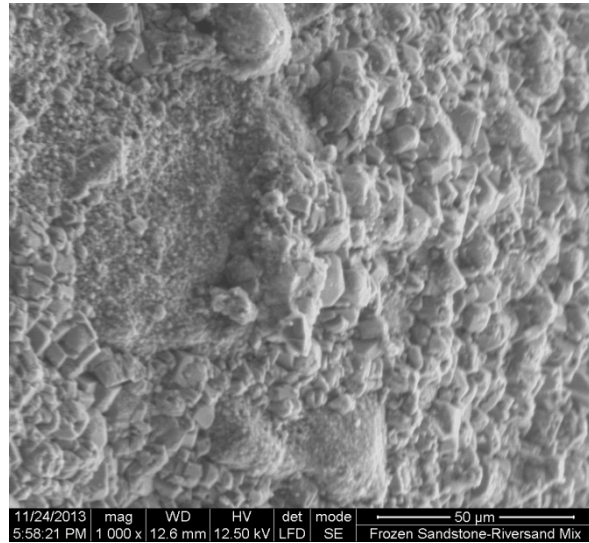
Limestone mix before freezing



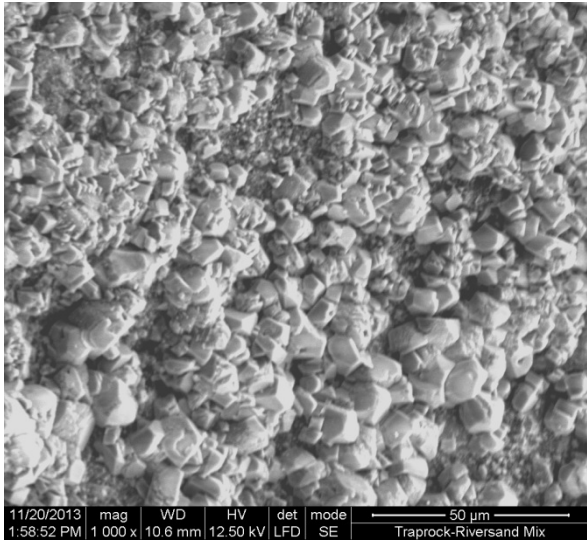
Limestone mix after freezing



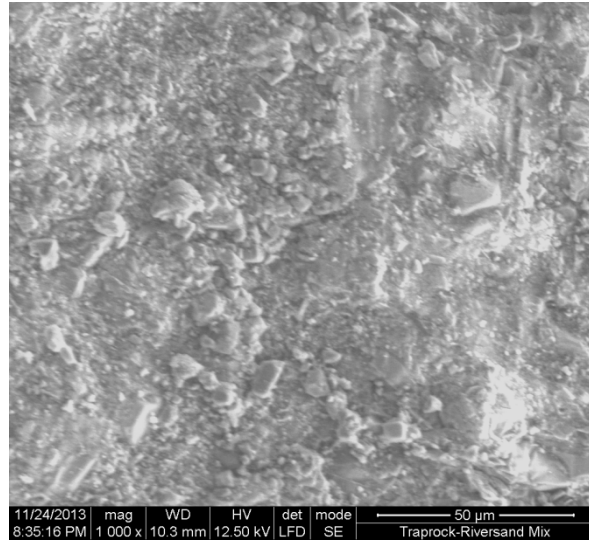
Sandstone mix before freezing



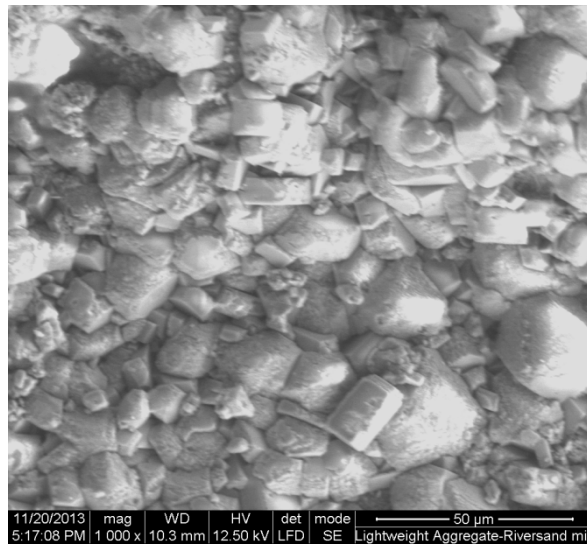
Sandstone mix after freezing



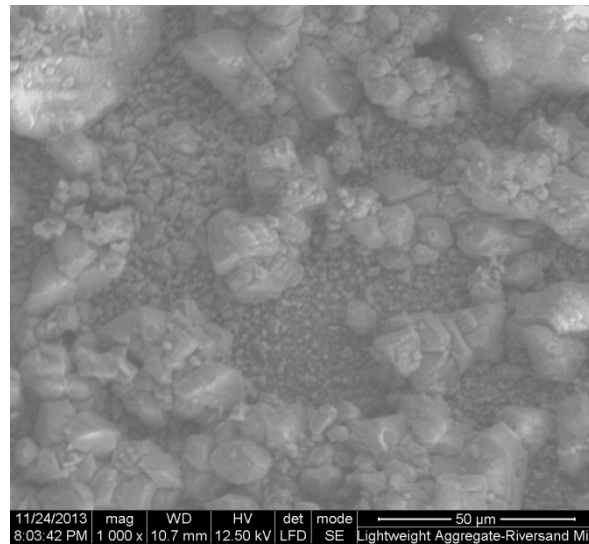
Trap rock mix before freezing



Trap rock mix after freezing

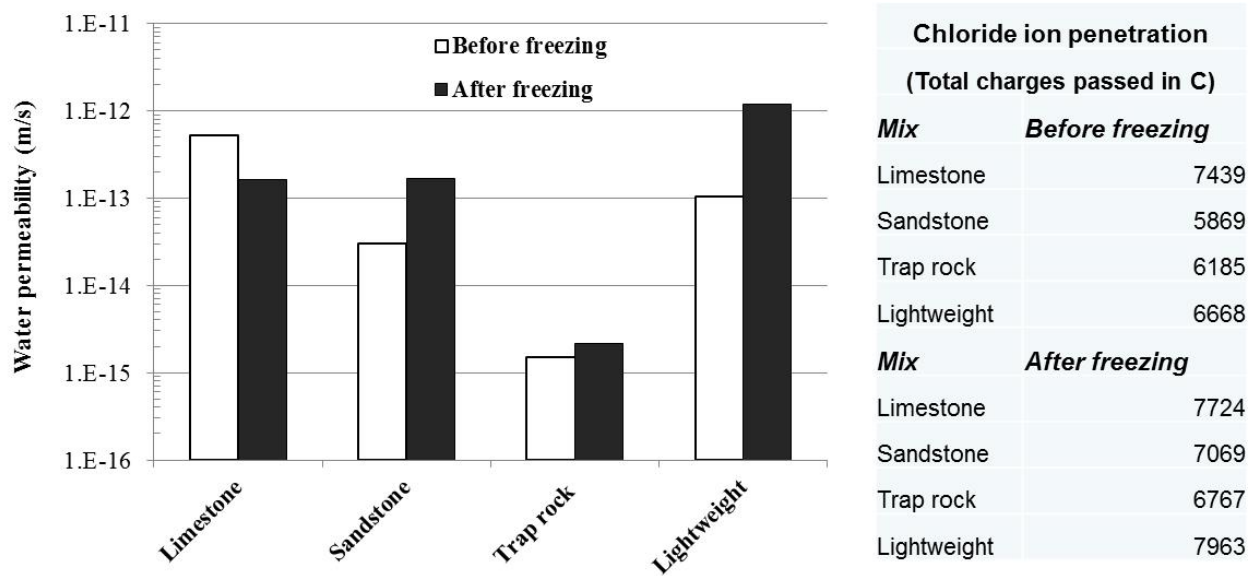


Lightweight mix before freezing



Lightweight mix after freezing

Selected Results – 28-day water and chloride permeability data



Water and chloride permeability results

- Formation of ice in concrete pores reduces permeability as it impedes liquid travel through the pore system¹. This is probably responsible for the decrease in permeability of the limestone mix.
- Microcrack formation increases permeability. This could have caused the increase in permeability in the other mixtures. Moreover, water and chloride permeability were measured after thawing of specimens and freeze-thaw cycling induces damage.

Preliminary Conclusions

- Conclusions cannot easily be made at this point by simply comparing SEM images with water and chloride permeability data due to variability in trends.
- However, it looks like images for limestone and trap rock mixes have morphology in some areas in the likeness of crystals, probably due to the contraction resulting from

freezing, and agglomeration of the constituents of cryogenic concrete, leading to blockage of pore spaces.

- ✓ Such morphology is more likely to resist cracking during cryogenic freezing due to restriction of water movement through interconnected concrete pores.
- The water and chloride permeability results somehow agree with the SEM image for trap rock. The mix showed the lowest water and chloride permeability values.
- These results would be compared to those of other tests, as part of the screening process. Thereafter, further mix designs would be considered in our search for concrete that resists damage during cryogenic freezing.

References

- [1] Kogbara, R. B., Iyengar, S. R., Grasley, Z. C., Masad, E. A., and Zollinger, D. G., “A review of concrete properties at cryogenic temperatures: Towards direct LNG containment,” *Constr. Build. Mater.*, 47, 760-770 (2013).
- [2] Krstulovic-Opara, N., “Liquefied natural gas storage: Material behavior of concrete at cryogenic temperatures,” *ACI Mater. J.*, 104(3), 297 – 306 (2007).
- [3] BSI, [BS EN 12390-8. Testing hardened concrete. Depth of penetration of water under pressure] British Standards Institution, London (2009).
- [4] Kogbara, R. B., Al-Tabbaa, A., and Iyengar, S. R., “Utilisation of magnesium phosphate cements to facilitate biodegradation within a stabilised/solidified contaminated soil,” *Water, Air, & Soil Pollution*, 216(1-4), 411-427 (2011).
- [5] Kogbara, R. B., Al-Tabbaa, A., Yi, Y., and Stegemann, J. A., “pH-dependent leaching behaviour and other performance properties of cement-treated mixed contaminated soil,” *Journal of Environmental Sciences*, 24(9), 1630 - 1638 (2012).
- [6] Kogbara, R. B., Yi, Y., and Al-Tabbaa, A., “Process envelopes for stabilisation/solidification of contaminated soil using lime-slag blend,” *Environmental Science and Pollution Research*, 18(8), 1286-96 (2011).