



Preliminary Investigation on the Development and Performance of Self-immune and Self-healing Soil-Cement Systems Under Freeze-Thaw Cycles

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Abstract. Inspiration from biological systems has recently fuelled research in the built environment to develop smart materials that comprise sustainable and resilient systems, which similarly can continually adapt and respond to their environment. Many of these activities have so far focused on concrete for structural applications. However, to date there has been little work on the development of effective smart systems for geotechnical applications. Those systems and relevant geotechnical applications pose very different and more difficult and challenging problems compared to concrete and require a complete rethink of how to design such smart systems. The focus of the work reported in this paper is on the development and performance of self-immune and self-healing soil-cement systems subject to freeze-thaw (f-t) cycles. This was addressed with two types of microcapsules: SikaAer[®] Solid and Lambson microcapsules. Unconfined compressive strength (UCS), water content and volume stability were investigated after f-t cycles. By adding 1% (by soil mass) Lambson microcapsule, the UCS of 20% cement stabilised soil samples subjected 1–12 f-t cycles was improved by 21–40%. The f-t durability was also largely improved by adding 1% (by soil mass) SikaAer[®] Solid, where no deterioration in UCS was observed; change in volume and moisture content was largely reduced; and no crack formation was observed by optical microscopy after 10 f-t cycles. This study presents evidence of the significant potential for soil-cement system to possess self-immune or self-healing capabilities through the implementation of appropriate microcapsules.

Keywords: Soil-cement · Microcapsule · Freeze-thaw (f-t) cycles
Self-immune · Self-healing

1 Introduction

The cost of the maintenance and repair of our civil infrastructure is tremendous: it is estimated that approximately £200 billion at 2010 price is needed for the maintenance and renewal for transportation networks alone in the UK from 2011 to 2030 [1]; ~\$10 billion per year is spent on the maintenance and repair of railway bridges and tunnels in Japan [2] and ~\$320 billion per year is needed in the US to restore its infrastructure to

a satisfactory condition [3]. Soil-cement systems are widely adopted in engineering practise, for example grouting applications, cut-off walls, embankments, pavements, and slopes. In cold regions, the freeze-thaw (f-t) condition is crucial for designing geotechnical infrastructure as many geotechnical structures undergo at least one f-t cycle annually, and many experience extensive repeated cycling. Deteriorations due to f-t cycles, result in civil infrastructures being out of service unpredictably. A soil-cement system will naturally be much more vulnerable to the degradation and weathering, e.g. f-t cycles, than concrete since the volume fraction and interspace of internal pores are much larger. Although many studies have suggested that soil-cement systems are more resistant to f-t than unstabilised soils, they still suffer significant degradation [4–8]. For example, the unconfined compressive strength (UCS) of 10% cement stabilised soil was reduced by up to 95% after 12 f-t cycles [8]. As the damage induced by f-t cycles usually exists inside the soil-cement systems, mitigation and repair are less possible, and sometimes the only way of maintenance is to rebuild the whole structure which is very costly. Currently, this issue is addressed either through initial overdesign, implementation of maintenance programs, or both [9]. However, those measures are uneconomical and time-consuming and therefore development of a soil-cement system with high f-t durability is much needed.

Inspiration from biological systems has recently fuelled research in the built environment to develop sustainable and resilient systems comprising materials that, similarly can continually adapt and respond to their environment. Much of these activities have so far focused on concrete for structural applications. Various healing agents and microcapsules were applied in cementitious material and a considerable recovery of mechanical properties as well as crack closure were reported by many researchers [10–12]. For example, ~95% crack sealing and ~25% strength recovery in 28 days was reported for mortars embedded with encapsulated expansive powder minerals (magnesium oxide, bentonite and quicklime), whose cracks were 0.4–0.5 mm [12]. However, to date there has been little work on applications to soil-cement systems. Those systems and relevant applications pose very different and challenging problems compared to concrete and require a complete rethink of how to design such self-healing systems. In this paper, the concepts of self-immune and self-healing are firstly applied in the soil-cement systems and investigated by a detailed experimental framework. The performance of self-immune and self-healing soil-cement systems using two types of microcapsules were accessed by UCS, volume stability and moisture content subject to f-t cycles. Visualisation of the formation of cracks was achieved using optical microscopy.

2 Materials and Methods

2.1 Materials

The materials used in this study were sand, CEM-I 52.5, Polwhite E China clay. The microcapsules used in this study include Lambson and SikaAer[®] Solid microcapsules supplied by Lambson Limited, UK and Sika Deutschland GmbH, respectively. SikaAer[®] Solid are microcapsules with size range of ~5–100 μm consisting of

prefabricated air bubbles with an elastic acrylonitril-polymer envelope, so they can provide a controlled air-entrainment. Lambson microcapsules, whose shell material is gelatine/gum Arabic blend, are microcapsules with an average size of $\sim 200\text{--}300\ \mu\text{m}$ that encapsulate sodium silicate/oil emulsion. Figure 1 shows the images of SikaAer[®] Solid and Lambson microcapsules observed by optical light microscope.

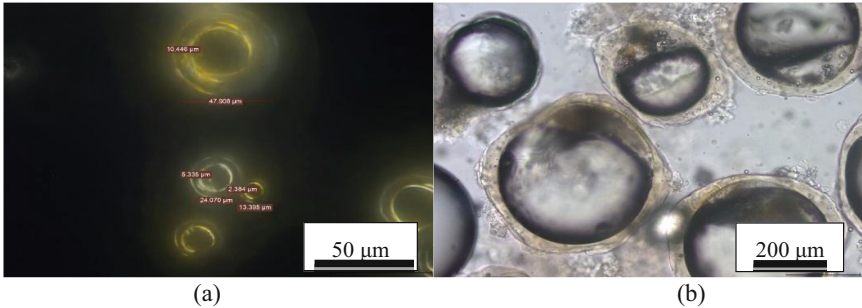


Fig. 1. The microcapsules used in the study (a) SikaAer[®] Solid microcapsules and (b) Lambson microcapsules

2.2 Sample Preparation and Testing Program

The artificial soil manufactured in this study composes of 85% sand and 15% clay. According to the unified soil classification system (USCS), the soil used can be classified as a clayey sand. Raw materials including sand, clay, cement, water and microcapsules were mixed in an automatic mixer. Four different mix designs were prepared and details of mixing proportions are summarised in Table 1.

Table 1. Compositions of soil-cement mixes

Mix ID	Mix ingredients ratios/mass (%)				Dry density (kg/m ³)	Agent
	Sand	Clay	Cement	Water		
C1	85	15	20	25	1760–1800	–
L1	85	15	20	25	1730–1750	Lambson microcapsules (1%)
C2	85	15	15	25	1720–1750	–
S1	85	15	15	25	1520–1570	SikaAer [®] Solid (1%)

Note: all the percentage in this table are proportional to the total mass of soil solid

A constant mixing time of 10 min was used to control the degree of mixing. The mixtures were placed in plastic cylindrical moulds with a height of 100 mm and an inner diameter about 50 mm. Samples were moulded in 3 layers and each layer were oscillated for 2 min in order to provide a uniform, adequate and similar compaction. All specimens were mixed in a standard laboratory environment with a temperature of

21 °C (± 2 °C) and 50% ($\pm 10\%$) relative humidity (RH) and cured for 7 days before subject to f-t cycles. Triplicate samples were prepared for each case to ensure repeatability.

The f-t curing were conducted as per ASTM: D560/D560 M-15 [13]. Each f-t cycle consists of 24 h of freezing at -25 °C (± 1 °C) and 23 h of thawing at 21 °C (± 2 °C) and 97% ($\pm 3\%$) RH. During both freezing and thawing, samples were placed on absorbent pads and free potable water was made available under the absorbent pads. For mixes C1 and L1, after each f-t cycle, they were left for 7 days for the self-healing process to develop. The f-t durability was then tested in terms of UCS, water content and volume stability. UCS was determined in triplicate based on ASTM: D4219 – 08 [14] using Uniframe 70-T0108/E Control Machine. A constant axial strain of 1% per minute until failure was applied.

Surface analysis of the exposed specimens using light optical microscopy can provide insight on the damage formation during f-t exposure by comparing samples with and without microcapsules. Cylindrical disk specimens with 10 mm thick and 50 mm diameter were prepared for this purpose. A Leica DM2700 upright optical microscope was used to observe crack distribution induced by the f-t cycles on soil-cement samples.

3 Results and Discussion

3.1 UCS

The incorporation of the SikaAer[®] Solid microcapsules aims to build a self-immune soil-cement system by providing controlled air-entrainment. Compared with conventional air-entrainment agents, SikaAer[®] Solid microcapsules occupy space within the soil matrix and therefore they do not increase the void ratio of the soil. Meanwhile, they do not break during the f-t cycles so they create room for the water to expand and contract since their elastic shells with the air inside are compressible. In addition, since their shells are impermeable to water, SikaAer[®] Solid microcapsules do not increase the capillary suction and the potential to absorb water. Figure 2 presents the variation in UCS of C2 and S1 samples after 10 f-t cycles. It can be seen that the initial strength was reduced by $\sim 24\%$ by adding 1% of SikaAer[®] Solid. However, the UCS of S1 samples increased with the number of f-t cycles while the UCS of C2 samples dropped rapidly. After 5 f-t cycles, the UCS of S1 sample was considerably higher than that of C2 sample. Increase in UCS by $\sim 33\%$ was observed for S1 sample due to the continuous cement hydration while reduction of $\sim 82\%$ was observed for C2 sample after 10 f-t cycles due to the damaging effect of water expansion.

Figure 2 shows that the L1 samples containing 1% Lambson microcapsules exhibited similar initial UCS with the C1 control samples. However, it is noticeable that after 1 f-t cycle and 7 days healing, the UCS of L1 samples were 40% higher than the C1 samples. This indicates that the Lambson microcapsules were broken after f-t exposure and the sodium silicate solution within them was released to enhance continuous hydration. More C-S-H gel were produced as sodium silicate reacts with calcium hydroxide produced from cement hydration [10, 11]. The UCS of L1 samples

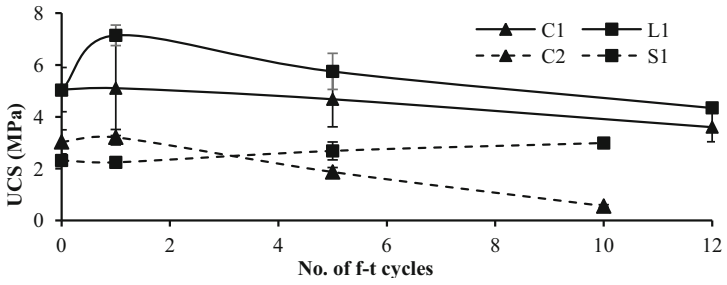


Fig. 2. UCS of soil-cement samples vs. f-t cycles

after 5 and 12 f-t cycles and self-healed for 7 days were 23% and ~21% higher than the C1 samples, respectively. UCS of control samples gradually reduced with the increase of f-t cycles whilst that of Lambson microcapsules embedded samples were higher than its initial UCS even after 5 f-t exposure. The results indicate that the addition of Lambson microcapsules provided excellent self-healing ability for soil-cement samples. The most effective healing was achieved at the first cycle and was effective even after 12 cycles.

3.2 Volume and Water Content

Many studies reported that the volume of soil/soil-cement systems increased with an increasing amount of f-t cycles [16], [17, 18]. As shown in Fig. 3a, the volume of C2 samples increased as the number of f-t cycles increased, where a change of up to ~11% was observed after 10 cycles. By adding SikaAer® Solid, the volumetric change of soil-cement system (sample S1) was largely reduced and only <2% volumetric change was measured after 10 f-t cycles. It indicates that the encapsulated air bubbles inside the matrix compensated the volumetric change caused by the volume expansion and contraction during the phase change of water. Increasing the cement content significantly reduced the volume change while the Lambson microcapsules had little effect on the volumetric change compared to the control sample (C1) after up to 12 f-t cycles. The moisture contents of all the 4 mixes increased with the f-t cycles (Fig. 3b).

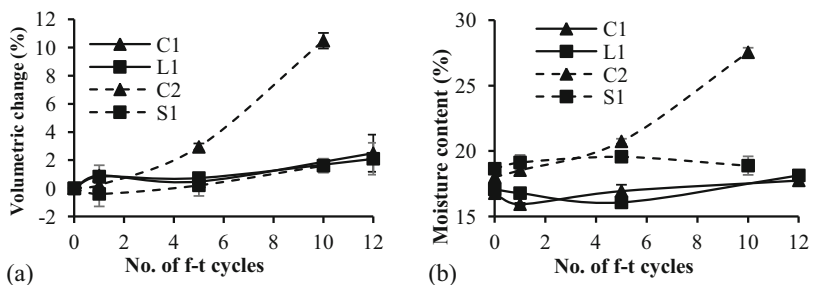


Fig. 3. (a) Volumetric change and (b) Water content of samples after f-t cycles

It is noticeable that the increase in moisture content after f-t cycles was largely reduced by adding SikaAer® Solid. After 10 f-t cycles, the moisture content of C2 increased from 18% to 27.5% while the moisture content of S1 changed slightly at 18.5–19.5%. The increase moisture content for C1 was caused by the continuous water absorption and pore expansion during f-t process. Addition of SikaAer® Solid reduced the pore expansion during freezing by providing expansion space therefore the capillary water absorption was also reduced. As for the L1 samples, the moisture content reduced before it kept increasing after 5 f-t cycles while the moisture content of C1

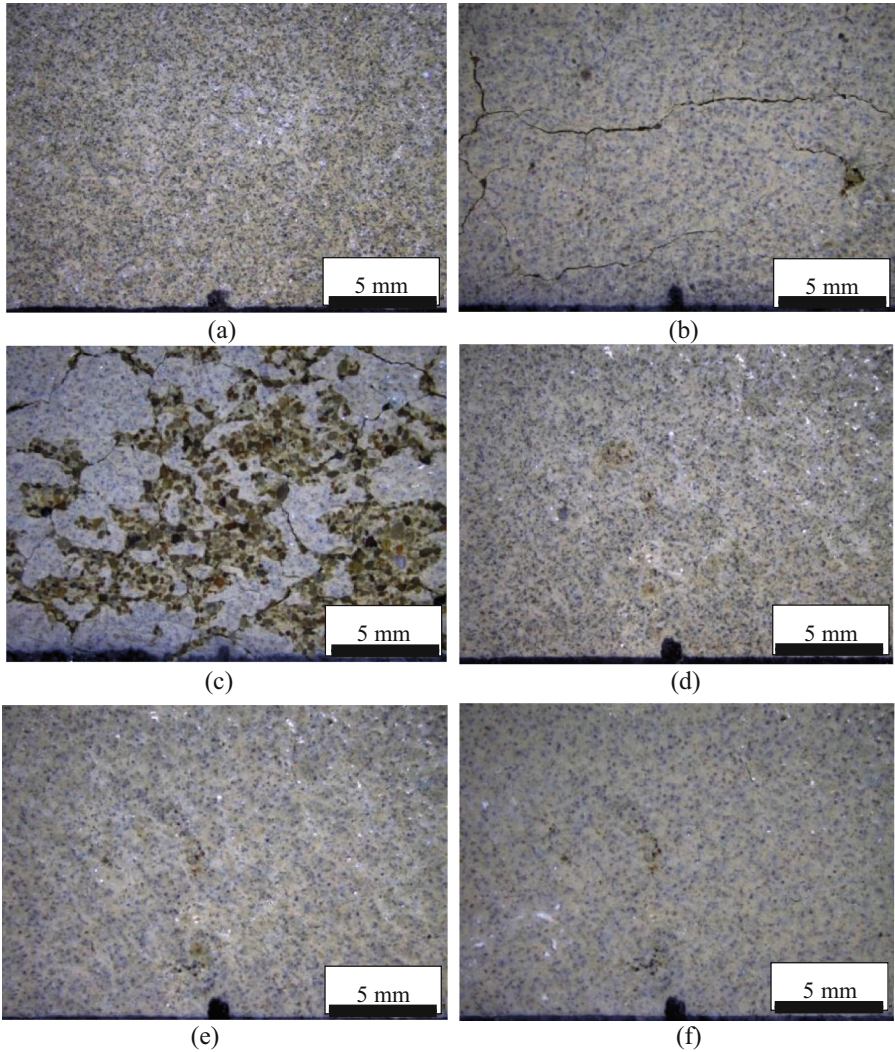


Fig. 4. C2 samples after (a) 0, (b) 5 and (c) 8 f-t cycles and S1 samples after (d) 0, (e) 5 and (f) 10 f-t cycles

samples kept increasing after 1 f-t cycle. It is noted that the moisture content of L1 was higher than that of C1 after 12 cycles. This can be explained by the fact that the use of Lambson microcapsules will leave voids within the matrix after breakage thus increase the void ratio, leading to higher potential to absorb water. The variation of water content correlated well with the volumetric change, indicating the volumetric change is highly related to the absorption of water. In addition, samples have higher UCS generally exhibited lower volumetric change and lower increase in moisture content.

3.3 Optical Microscopy

The surface conditions of the 10 mm thick C2 and S1 samples before and after f-t exposure are presented in Fig. 4.

Damages and cracks can be seen on the surface of C2 samples after f-t cycles while no crack was observed for S1 samples even after 10 f-t cycles. It is believed that the self-immune ability of soil-cement system was provided by the embedded compressible SikaAer[®] Solid.

4 Conclusions

In this study, performance of cement-treated clayey sand embedded with two types of microcapsules was evaluated after freeze-thaw (f-t) exposure. It was found that the Lambson microcapsules improved the self-healing ability of soil-cement samples with an increase of 21–40% in UCS after 12 cycles of f-t and the biggest improvement was observed after the first f-t. This indicated that the Lambson microcapsules ruptured after f-t exposure and the sodium silicate solution within them was released to enhance self-healing. The SikaAer[®] Solid substantially improved the f-t durability of soil-cement by providing controlled air-entrainment building a self-immune system. They created room for the water to expand and contract with the soil-cement matrix during the f-t process. After 10 f-t cycles, the UCS increased by ~33% for samples embedded with SikaAer[®] Solid while a reduction of ~82% was observed for samples without the microcapsules. The change in volume and moisture content after f-t cycles were both largely reduced. Optical microscopy showed no cracking on the surface of samples embedded with SikaAer[®] Solid while many cracks and damages were captured on the surface of control samples. In this paper, the concepts of self-immune and self-healing microcapsules are the first to be applied in the soil-cement systems and provide new perspectives to address f-t problems. Preliminary investigation on their performance under f-t cycles presents initial evidence of the significant potential for soil-cement system to possess self-healing or self-immune capabilities through the implementation of appropriate microcapsules.

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References

1. Mills, J., Shilson, S., Woodley, Q., Woodwark, A.: Keeping Britain moving, United Kingdom's Transport Infrastructure Needs (2011). http://www.mckinsey.com/~ /media/McKinsey/ dotcom/client_service/Infrastructure/PDFs/Keeping_Britain_Moving_the_United_Kingdoms_ Transport_Infrastructure_Needs.ashx
2. Ahn, T.-H., Kishi, T.: Crack self-healing behavior of cementitious composites incorporating various mineral admixtures. *J. Adv. Concr. Technol.* **8**, 171–186 (2010). <https://doi.org/10.3151/jact.8.171>
3. Natale, P.J.: Failing Infrastructure—Threatening our Economy and Way of Life, NJIT Magazine (2010)
4. Altun, S., Sezer, A., Erol, A.: The effects of additives and curing conditions on the mechanical behavior of a silty soil. *Cold Reg. Sci. Technol.* **56**, 135–140 (2009). <https://doi.org/10.1016/j.coldregions.2008.11.007>
5. Davis, K., Warr, L., Burns, S., Hoppe, E.J.: Physical and chemical behavior of four cement-treated aggregates. *J. Mater. Civ. Eng.* **19**, 891–897 (2007). [https://doi.org/10.1061/\(ASCE\)0899-1561\(2007\)19:10\(891\)](https://doi.org/10.1061/(ASCE)0899-1561(2007)19:10(891))
6. Qi, J., Ma, W., Song, C.: Influence of freeze-thaw on engineering properties of a silty soil. *Cold Reg. Sci. Technol.* **53**, 397–404 (2008). <https://doi.org/10.1016/j.coldregions.2007.05.010>
7. Wang, D.Y., Ma, W., Niu, Y.H., Chang, X.X., Wen, Z.: Effects of cyclic freezing and thawing on mechanical properties of Qinghai-Tibet clay. *Cold Reg. Sci. Technol.* **48**, 34–43 (2007). <https://doi.org/10.1016/j.coldregions.2006.09.008>
8. Jamshidi, R.J., Lake, C.B., Barnes, C.L.: Examining freeze / thaw cycling and its impact on the hydraulic performance of a cement-treated silty sand. *J. Cold Reg. Eng. ASCE.* **29**, 04014014 (2015). [https://doi.org/10.1061/\(asce\)cr.1943-5495.0000081](https://doi.org/10.1061/(asce)cr.1943-5495.0000081)
9. Harbottle, M.J., Lam, M., Botusharova, S.P., Gardner, D.R.: Self-healing soil: biomimetic engineering of geotechnical structures to respond to damage. In: Proceedings of the 7th International Congress on Environmental Geotechnics (2014)
10. Kanellopoulos, A., Qureshi, T.S., Al-Tabbaa, A.: Glass encapsulated minerals for self-healing in cement based composites. *Constr. Build. Mater.* **98**, 780–791 (2015). <https://doi.org/10.1016/j.conbuildmat.2015.08.127>
11. Pelletier, M.M., Brown, R., Shukla, A., Bose, A.: Self-healing concrete with a microencapsulated healing agent. University of Rhode Island, Kingston, USA (2010)
12. Qureshi, T.S., Kanellopoulos, A., Al-Tabbaa, A.: Encapsulation of expansive powder minerals within a concentric glass capsule system for self-healing concrete. *Constr. Build. Mater.* **121**, 629–643 (2016). <https://doi.org/10.1016/j.conbuildmat.2016.06.030>
13. ASTM: D560/D560 M - 15, Standard Test Methods for Freezing and Thawing Compacted Soil-Cement Mixtures, ASTM International (2015). <https://doi.org/10.1520/d0560-03>
14. ASTM: D4219 - 08, Standard Test Method for Unconfined Compressive Strength Index of Chemical- Grouted Soils, ASTM International (2008). <https://doi.org/10.1520/d4219-08.2>
15. Liu, J., Wang, T., Tian, Y.: Experimental study of the dynamic properties of cement- and lime-modified clay soils subjected to freeze-thaw cycles. *Cold Reg. Sci. Technol.* **61**, 29–33 (2010). <https://doi.org/10.1016/j.coldregions.2010.01.002>
16. Shibi, T., Kamei, T.: Effect of freeze-thaw cycles on the strength and physical properties of cement-stabilised soil containing recycled bassanite and coal ash. *Cold Reg. Sci. Technol.* **106–107**, 36–45 (2014). <https://doi.org/10.1016/j.coldregions.2014.06.005>