**TRANSPORTATION POOLED FUND PROGRAM**

**QUARTERLY PROGRESS REPORT**

Lead Agency (FHWA or State DOT): Oklahoma Department of Transportation

**INSTRUCTIONS:**

*Project Managers and/or research project investigators should complete a quarterly progress report for each calendar quarter during which the projects are active. Please provide a project schedule status of the research activities tied to each task that is defined in the proposal; a percentage completion of each task; a concise discussion (2 or 3 sentences) of the current status, including accomplishments and problems encountered, if any. List all tasks, even if no work was done during this period.*

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| **Transportation Pooled Fund Program Project #***TPF-5(448)* | **Transportation Pooled Fund Program - Report Period:**O Quarter 1 (January 1 – March 31)X Quarter 2 (April 1 – June 30)O Quarter 3 (July 1 – September 30)O Quarter 4 (October 1 – December 31) |
| **Project Title:****Integrating Construction Practices and Weather Into Freeze Thaw Specifications** |
| **Name of Project Manager(s):****Tyler Ley** | **Phone Number:****405-744-5257** | **E-Mail**Tyler.ley@okstate.edu |
| **Lead Agency Project ID:****TPF-TPF5(448)** | **Other Project ID (i.e., contract #):**AA-1-501021 | **Project Start Date:**August 30, 2020 |
| **Original Project End Date:**August 30, 2023 | **Current Project End Date:****August 30,2023** | **Number of Extensions:**0 |

Project schedule status:

 X On schedule On revised schedule □ Ahead of schedule □ Behind schedule

Overall Project Statistics:

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|  **Total Project Budget** |  **Total Cost to Date for Project** |  **Percentage of Work**  **Completed to Date** |
| $660,000 | $140,000 | 22% |

***Quarterly*** Project Statistics:

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|  **Total Project Expenses**  **and Percentage This Quarter** |  **Total Amount of Funds**  **Expended This Quarter** |  **Total Percentage of**  **Time Used to Date** |
| 6% | $40,000 | 15% |

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| **Project Description**:Concrete can be damaged when it is 1) sufficiently wet (has reached a critical degree of saturation) and 2) is exposed to temperature cycles that enable freezing and thawing. The damage that occurs due to freezing and thawing can lead to premature deterioration, costly repairs, and premature replacement of concrete infrastructure elements. Current specifications for frost durability are largely based on work completed in the 1950s, and while this work included many landmark discoveries (Kleiger 1952, 1954). This work from the 1950s may not be representative of materials used in modern concrete mixtures. The objective of this work is to build on previous research efforts to produce improved specifications and advance existing test methods; while, improve the underlying understanding of freeze thaw damage. This work will specifically focus on construction practices and the impact of weather.The objectives are: 1. Quantify how different weather conditions impact the freeze thaw performance of concrete with low-cost data loggers. This work has been started under this existing project but these samples should be distributed in the field and used to quantify the combination of saturation and freeze thaw cycles in different states.
2. Investigate the freeze thaw performance of existing structures in different climates with different air void qualities. In combination with quantifying the weather in different environments, structures should be found in these structures with different quality of air void systems to determine how they perform. This will provide true case studies of field performance in a quantified exposure.
3. Expand the freeze thaw model to a larger range of mixtures to see if the trends still hold.
4. Further evaluation of the accuracy of the modeling predictions for determining the matrix saturation and the relationship between the secondary sorption and formation factor.
5. Better understand the damage propagation after critical saturation is reached.
6. Extension of this work to include salts such as those that result in calcium oxychloride to further improve the computational modeling predictions.
7. Determine how air void filling impacts the durability of concrete from freeze thaw cycles.
8. Develop freeze thaw specifications based on concrete quality, air void system, and local weather conditions.
9. Determine how construction methods such as pumping, mixing time, paving vibration, and hand held vibrators impact the air void spacing within concrete
10. Improve the SAM by making the measurement more consistent through developing a semi-automated testing procedure and improving reliability prediction.
11. Further refine a rapid test method that measures the uptake and fluid and resistivity of the concrete to determine the freeze thaw durability of concrete
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| **Progress this Quarter (includes meetings, work plan status, contract status, significant progress, etc.):**1. Quantify how different weather conditions impact the freeze thaw performance of concrete with low-cost data loggers. This work has been started under this existing project but these samples should be distributed in the field and used to quantify the combination of saturation and freeze thaw cycles in different states.

Data is continuing to be collected and analyzed. The analysis so far has focused on the data produced in Oklahoma. This will be completed in the next quarter and then the data will be extended to other regions. Laboratory testing is being completed to compliment the data collected on the weather boxes. The percentage of freezable solution is being measured using low differential scanning calorimetry [1]. The critical degree of saturation is being determined using length change measurements [2]. This experiment will aid in determining the depth of freeze thaw damage in the specimens. The impact of the temperature on the degree of saturation is being measured using Dynamic Vapor Sorption DVS Q5000. These laboratory experiments is being performed and will be finished in the fourth quarter of 2021. 1. Investigate the freeze thaw performance of existing structures in different climates with different air void qualities. In combination with quantifying the weather in different environments, structures should be found in these structures with different quality of air void systems to determine how they perform. This will provide true case studies of field performance in a quantified exposure.

Samples have been provided from Iowa for this. This will start later in the project. More projects are being sought for this.1. Expand the freeze thaw model to a larger range of mixtures to see if the trends still hold.

In part 1 of the project, researchers had developed a correlation between the probability of failure due to freeze-thaw cycles with respect to the degree of saturation of the mortar samples tested (Figure 1). The probability of failure increases with an increase of DOS in the sample. For example, all the samples with DOS higher than 88% develop FT damage. The failure region starts at DOS of ≈77.5% for the samples with a SAM number ≤0.20 compared to the DOS of ≈72.5% for the samples with a SAM number between 0.20 and 0.60. In addition, when the DOS of the sample is around the critical level of saturation (72.5% <DOS<88%), the probability of failure tends to be higher for samples with high SAM number (SAM > 0.20) compared to samples with a low SAM number (≤0.20). Ley et al. [3] showed that a SAM Number of 0.20 best correlates with the recommended spacing factor of 200 μm for FT durability (ACI 201.2R limit). The reduced quality of air void distribution explains the higher probability of failure in samples with a SAM number > 0.20. This is in accordance with the data collected in [4], where it was concluded that higher quality air-void systems, quantified by lower SAM numbers, may resist freeze-thaw damage at higher levels of saturation than those with poorly distributed air void systems.This correlation in Figure 1 was collected on 134 mortar samples prepared with cement type I/II and with different air void content and air void quality. Only 9 different mixtures were tested to obtain Figure 1. Figure 1. Probability of failure with respect to the degree of saturation [2]The research is expanding this plot by adding a number of samples from a much wider array of mixtures. This helps validate the SAM and also the importance of freeze thaw durability. These samples are being prepared for analysis and they will be added to Figure 1. The bin size for the statistical analysis in Figure 1 is 5%. These additional measurements will allow decreasing the bin size to 2% which will give more confidence in the correlation and conclusion drawn out of this graph.  1. Further evaluation of the accuracy of the modeling predictions for determining the matrix saturation and the relationship between the secondary sorption and formation factor.

In the previous part of the project, a correlation has been established between the apparent formation factor and the initial and secondary sorptivity coefficient of plain concrete samples [5, 6]. The correlation is not established yet for concrete samples with supplementary cementitious materials. The apparent formation factor as well as the initial and secondary sorptivity of concrete samples with varying fly ash content will be tested in the second part or the project [7]. The correlation will be determined for samples with fly ash and compared with the correlation obtained on plain concrete samples. This relationship provides a powerful tool in quality control to obtain *FAP* that relates to absorption properties by using a simple immersion test. The fluid absorption properties are key parameters in service life prediction of concrete structures subjected to freezing-and-thawing cycles. Concrete samples with 25 different mixture design has been prepared. The mixture design of these concrete samples is given in Table 1. Table . The mixture proportions and fresh properties of the cementitious samples (normalized to 1000kg/m3)

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| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Mixture | Coarse aggregate 1(kg/m3) | Coarse aggregate 2(kg/m3) | Sand (kg/m3) | Cement (kg/m3) | Fly ash(kg/m3) | Water (kg/m3) | AEA(g/m3) | Adva Cast 575(g/m3) | Air content (%),SAM number |
| 1 | 469.3 | 0.0 | 305.2 | 124.0 | 30.9 | 69.8 | 0.8 | 0.0 | 2.55, 0.350 |
| 2 | 468.4 | 0.0 | 304.6 | 123.8 | 30.9 | 69.6 | 2.7 | 0.0 | 5.40, 0.155 |
| 3 | 468.1 | 0.0 | 305.8 | 116.1 | 38.7 | 69.7 | 1.5 | 0.0 | 2.10, 0.660 |
| 4 | 466.1 | 0.0 | 304.4 | 115.6 | 38.5 | 69.4 | 6.0 | 0.0 | 7.15, 0.100 |
| 5 | 467.6 | 0.0 | 305.5 | 116.0 | 38.7 | 69.6 | 2.7 | 0.0 | 2.9, 0.200 |
| 6 | 468.1 | 0.0 | 305.8 | 108.5 | 46.5 | 69.8 | 1.3 | 0.0 | 1.80, 0.630 |
| 7 | 466.1 | 0.0 | 304.4 | 108.0 | 46.3 | 69.5 | 5.7 | 0.0 | 5.65, 0.130 |
| 8 | 467.5 | 0.0 | 305.4 | 108.4 | 46.4 | 69.7 | 2.6 | 0.0 | 3.2, 0.315 |
| 9 | 467.9 | 0.0 | 305.6 | 100.9 | 54.3 | 69.8 | 1.5 | 0.0 | 1.95, 0.545 |
| 10 | 465.8 | 0.0 | 304.3 | 100.4 | 54.1 | 69.5 | 5.9 | 0.0 | 7.10, 0.100 |
| 11 | 467.4 | 0.0 | 305.3 | 100.8 | 54.3 | 69.8 | 2.6 | 0.0 | 2.7, 0.24 |
| 12 | 469.5 | 0.0 | 304.5 | 93.1 | 61.9 | 69.8 | 1.1 | 0.0 | 2.55, 0.570 |
| 13 | 468.6 | 0.0 | 303.9 | 93.0 | 61.8 | 69.7 | 3.1 | 0.0 | 5.85,0.100 |
| 14 | 262.2 | 202.3 | 312.2 | 112.7 | 28.2 | 63.4 | 1.1 | 18.0 | 5.75, 0.24 |
| 15 | 261.9 | 202.0 | 311.8 | 112.5 | 28.1 | 63.3 | 2.2 | 18.0 | 8.5, 0.065 |
| 16 | 262.5 | 202.5 | 312.5 | 112.8 | 28.2 | 63.5 | 0.7 | 17.3 | 3.6, 0.505 |
| 17 | 260.4 | 202.9 | 313.0 | 105.9 | 35.3 | 63.6 | 0.7 | 18.1 | 4.9, 0.4 |
| 18 | 260.2 | 202.7 | 312.7 | 105.8 | 35.3 | 63.5 | 1.7 | 18.2 | 7.3, 0.115 |
| 19 | 259.3 | 203.0 | 313.2 | 98.9 | 42.4 | 63.6 | 1.6 | 18.0 | 5.6, 0.17 |
| 20 | 259.7 | 203.2 | 313.6 | 99.0 | 42.4 | 63.7 | 0.8 | 17.6 | 4.150.395 |
| 21 | 259.8 | 202.1 | 313.8 | 92.0 | 49.5 | 63.7 | 1.8 | 17.4 | 5.40.18 |
| 22 | 259.4 | 201.8 | 313.3 | 91.9 | 49.5 | 63.6 | 1.5 | 18.9 | 4.3, 0.38 |
| 23 | 259.0 | 202.4 | 314.3 | 85.1 | 56.7 | 63.8 | 0.7 | 17.9 | 2.75, 0.5 |
| 24 | 258.6 | 202.1 | 313.9 | 85.0 | 56.6 | 63.7 | 2.3 | 17.7 | 6.5, 0.21 |
| 25 | 258.7 | 202.2 | 314.0 | 85.0 | 56.7 | 63.7 | 1.8 | 18.0 | 4.65, 0.325 |

The apparent formation factor measurements have been performed using two concrete samples from each mixture design. For this measurement, the uniaxial resistance was measured using AASHTO TP 119 [8] after 7 and 14 days of immersion in the simulated pore solution (Option A). After the different duration of immersion, the resistance was measured along with temperature, and sample geometry. The resistivity of the specimen was calculated using equation 1.

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| $$ρ\_{s}=R⋅\frac{A}{L}$$ |  |

Where, $ρ\_{s} $is theresistivity of specimen, $R$ is the resistance of the specimen (Ω), $A$ is specimen cross-sectional area (m2), $L$ = average specimen length (m). Temperature corrections were made using the Arrhenius approach following the guidance of Coyle et al. [9] with an activation energy of 15 kJ/mol.The formation factor was calculated using equation 2

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| $$F=\frac{ρ\_{s}}{ρ\_{ps}}$$ |  |

Where, the resistivity of the simulated pore solution (𝜌𝑝s) was equal to 0.127 Ω.mFor the absorption test, from one of these concrete samples, 3 slices of 2 inches thickness each were cut from the middle section of the sample and were put at 50% RH environment and 23°C in order to reach equilibrium. The slices are currently in this 50% RH environment and once they reach equilibrium their initial and secondary sorptivity will be determined according to ASTM 1585 [7]. A correlation will then be established between these values and the apparent formation factor values. The data are predicted to be available to share in the second quarter of 2022 and that depends on the time needed for the samples to reach equilibrium. 1. Better understand the damage propagation after critical saturation is reached.

X-ray computed tomography will be used to measure the FT damage in mortar samples with varying degrees of saturation. These results will be correlated with the probability of failure obtained on mortar samples with different DOS (Figure 1). Samples have been prepared at Oregon State and sent to Oklahoma State for testing. The samples have been conditioned and scanned and data is being generated. The results so far are very promising. 1. Extension of this work to include salts such as those that result in calcium oxychloride to further improve the computational modeling predictions.

In the first part of the project, researchers have studied the salt damage that developed in mortar samples due to the formation of calcium oxychloride. Mortar samples with varying air content, varying air void quality and varying fly ash content were saturated in 20% calcium chloride (CaCl2) solution. Micro X-ray fluorescent spectroscopy was used to determine that the chloride ions were uniformly distributed throughout the sample. Saturated samples were exposed to temperature cycles varying from 50°C to 5°C while being immersed in 20% CaCL2 solution. During the cooling period, calcium oxychloride (CaOXY) develops in the pores of the mortar samples. During the heating period, CaOXY melts. CaOXY is a product of the reaction between CaCl2 and calcium hydroxide. The volume of CaOXY is smaller than the reactants. Consequently, during the cooling period, due to the volume shrinkage induced by CaOXY formation, 20% CaCl2 solution can diffuse and refill the pores of the cementitious materials. During the heating process, CaOXY melts and expand in volume leading thus to internal pressure and salt damage. The length of the sample was measured at the beginning and end of each temperature cycle using a high precision micrometer. When a sample is damaged, an increase in its length will be measured. The residual strain was used as an indicator for damage and was calculated according to equation 1.

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|  | $$Residual strain= \frac{l\_{i}-l\_{0}}{l\_{0}}$$ | (1) |

Where, l0 is the initial length of the sample, li is the length of the sample after each temperature cycle. The data collected show that * Higher fly ash content mixtures (35% and 40%) did not develop damage regardless of the air void content. This can be explained by the fact that the calcium hydroxide content is not high enough to generate sufficient CaOXY to lead to damage [10-12].
* Lower fly ash content mixtures (0-20%) developed salt damage irrespective of the air void content. This can be explained by the fact that the calcium hydroxide content is high enough that a volume of CaOXY exceeds the air void volume resulting in damage.
* Intermediate fly ash content mixtures (25% and 30%) demonstrated that samples with higher air content had improved resistance to salt damage (compared to those with a lower entrained air content). This can be explained by the difference in the available space that the air voids provide for CaOXY to form.
* The absorption of the fluid by the samples during temperature cycling has a signifincant impact on increasing salt damage development
1. Determine how air void filling impacts the durability of concrete from salt damage.

Differential scanning calorimetry (DSC) is an experimental technique in which the difference in the amount of heat required to increase the temperature of a sample compared to a reference is measured as a function of the temperature. This technique can be used to determine the phase change in the sample as well as quantity of material undergoing the phase change. A powder will be prepared from each mix design tested in task 6 (salt damage) and will be mixed with 20% CaCl2 solution at a ratio of 4:1 [19]. The CaOXY that develops will be quantified. The volume of calcium oxychloride will then be concluded.X-ray CT scans will be conducted to measure the filling of voids due to CaOXY (formation and melting). The measurements obtained from X-ray CT will be compared with the volume of calcium oxychloride measurements using the LT-DSC The CT scans are being completed and air void filling is being observed. This shows that this is an important mechanism in the deterioration of concrete.1. Develop freeze thaw specifications based on concrete quality, air void system, and local weather conditions.’

This will be done at the end of the project. 1. Determine how construction methods such as pumping, mixing time, paving vibration, and hand held vibrators impact the air void spacing within concrete

OK state is looking at vibration and how it impacts the air void system in concrete. Some of this is to improve the SAM and the accuracy with low slump concrete. Some of this is with field concrete and with different vibration. We are also going to do some field mixing time measurements. This is still in the planning stages.1. Improve the SAM by making the measurement more consistent through developing a semi-automated testing procedure and improving reliability prediction.

A new gauge has been developed and it includes the error algorithm within it. The gauge is being made more robust and is being tested in the laboratory to ensure that it is accurate. The team has worked on automating different parts of the SAM but more work is needed. The states will be provided new gauges as soon as they are done with testing. This should occur in the next quarter. 1. Further refine a rapid test method that measures the uptake and fluid and resistivity of the concrete to determine the freeze thaw durability of concrete

In this research study, the authors have been determining the critical degree of saturation for different mixtures. In addition for some of these mixtures they will be measuring the formation factor and correlating it with the sorptivity coefficient. Consequently, they will work on finding a correlation between the second sorptivity coefficient value and the critical degree of saturation. As described in section 4, the apparent formation factor has been calculated on concrete samples with 25 different mixture designs and samples are being preconditioned in order to test their sorptivity coefficient. **References:** 1. Ghantous, R.M. and J. Weiss, Does the water to cement ration of concrete impact the value of its critical degree of saturation? , in 10th Inter national 14 Conference on Fracture Mechanics of Concrete and Concrete Structures. 2019: Bayonne, France. p. 1-10.2. Ghantous, R.M., et al., Determining the freeze-thaw performance of mortar samples using length change measurements during freezing. accepted in cement and concrete composite 2020.3. Ley, M.T., et al., Determining the air-void distribution in fresh concrete with the Sequential Air Method. Construction and Building Materials, 2017. **150**: p. 723-737.4. Todak, H.N., Durability assessments of concrete using electrical properties and acoustic emission testing, in School of Civil Engineering. 2015, Purdue University: West Lafayette. p. 143.5. Khanzadeh Moradllo, M., et al., Quantifying fluid filling of the air voids in air entrained concrete using neutron radiography. Cement and Concrete Composites, 2019. **104**.6. Moradllo, M.K., et al., Relating the formation factor of concrete to water absorption. ACI Mater. J., 2018. **Submitted**.7. ASTM C1585-13 Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes. 2013, ASTM International: West Conshohocken, PA.8. AASHTO, Standard Method of Test for Electrical Resistivity of a Concrete Cylinder Tested in a Uniaxial Resistance Test, in TP 119-20. 2020, American Association of State Highway and Transportation Officials: Washington DC.9. Coyle, A.T., et al., Comparison of linear temperature corrections and activation energy temperature corrections for electrical resistivity measurements of concrete. Advances in Civil Engineering Materials, 2018. **7**(1): p. 174-187.10. Suraneni, P., et al., Use of fly ash to minimize deicing salt damage in concrete pavements. Journal of the Transportation Research Board, 2017. **2629**: p. 24-32.11. Suraneni, P., et al., Role of supplementary cementitious material type in the mitigation of calcium oxychloride formation in cementitious pastes. Journal of Materials in Civil Engineering, 2018. **30**: p. 1-10.12. Suraneni, P., et al., Calcium oxychloride formation potential in cementitious pastes exposed to blends of deicing salt. ACI Materials Journal, 2017. **114**(4): p. 631-641.13. Del Mar Arribas-Colón, M., et al., Investigation of Premature Distress Around Joints in PCC Pavements: Parts I & II. 2012, Joint Transportation Research Program, Indiana Department of Transportation and Purdue University, West Lafayette, Indiana: Publication FHWA/IN/JTRP-2012/25 & FHWA/IN/JTRP-2012/26.14. Jones, W., et al., An Overview of Joint Deterioration in Concrete Pavement: Mechanisms, Solution Properties, and Sealers. 2013: West Lafayette, Indiana.15. Castro, J., et al., Durability of saw-cut joints in plain cement concrete pavements. 2011, Purdue University. Joint Transportation Research Program.16. Graveen, C., et al., Performance Related Specifications (PRS) for Concrete Pavements in Indiana, Volume 2: Technical Report. 2009.17. Engineers, A.S.o.C. ASCE 2017 infrastructure report card. 2017.18. Olek, J., M. Radlinski, and M. del Mar Arribas. Premature deterioration of joints in selected Indiana portland cement concrete pavements. 2007.19. Suraneni, P. and J. Weiss, Extending Low-Temperature Differential Scanning Calorimetry from Paste to Mortar and Concrete to Quantify the Potential for Calcium Oxychloride Formation. Advances in Civil Engineering Materials, 2018. **7**(1): p. 1-16. |
| **Anticipated work next quarter**:Continue to work on each task and hold biweekly meetings. |

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| **Significant Results:** |
| **Circumstance affecting project or budget. (Please describe any challenges encountered or anticipated that** **might affect the completion of the project within the time, scope and fiscal constraints set forth in the agreement, along with recommended solutions to those problems).** |

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| **Potential Implementation:**  |