



Transportation Infrastructure Precast Innovation Center (TRANS-IPIC)

University Transportation Center (UTC)

*Unveiling synergistic effects of Nano-modification and CO₂ curing on the
durability and carbon footprint of precast elements
PU-23-RP-02*

Quarterly Progress Report
For the performance period ending 09/30/2024

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N/A

Submitted to:

TRANS-IPIC UTC

University of Illinois Urbana-Champaign

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TRANS-IPIC Quarterly Progress Report:

Project Description:

1. Research Plan - Statement of Problem

The optimization of the application of new technologies and novel materials in precast concrete elements plays a crucial role in advancing the precast industry toward a greener future. The enhancement of durability and the reduction of carbon footprint of precast concrete elements are two of the main paths towards this advancement.

Both CO₂ curing (a precast treatment) and the use of nano-additives enhance the strength and reduce the porosity of cementitious composites, enhancing the durability of the concrete elements. However, CO₂ curing and nano-modification may interfere with each other if used simultaneously, especially considering that the addition of nanoparticles may affect the size of calcium hydroxide crystals, which react with the CO₂ during the CO₂ curing process. Thus, understanding the interactions between these two treatments is vital to produce superior quality precast concrete elements in terms of durability and sustainability.

2. Research Plan - Summary of Project Activities (Tasks)

To achieve the objectives of Phase I of this project, a systematic research approach will be followed, consisting of the following tasks:

Task 1. Characterization of the materials. This task will involve the physical and chemical characterization of the nanoparticles, the aggregates, and the cement to be used in this study, according to the relevant standards for each type of the proposed material. Particle size, oxide and mineralogical analyses will be performed.

Task 2. Preparation of specimens and curing process. Concrete, mortar and cement paste mixtures with 0%, 0.5%, 1%, and 2% of nanoparticles by mass of cement and two different water-to-cement ratios will be used in this study. Two different nanomaterials will be used: nano-silica and carbon black. Thus, a total of 14 different mixtures will be studied for each cementitious composite (paste, mortar, and concrete). Various types of specimens will be prepared and used to perform microstructural analysis, chemical analysis, and to determine the compressive strength development, transport properties and durability performance. Two different curing regimes and two different curing times will be examined: (i) standard curing at 21 ± 1 °C and $50\% \pm 5\%$ RH (for reference) for 12 hours, (ii) CO₂ curing (20% concentration) for 12 hours (from age 24h to 36h) at a temperature of 23 ± 1 °C and $50\% \pm 5\%$ RH.

Task 3. Analysis of hydration process, porosity and microstructure.

Image analysis of microstructure of concrete, captured via optical microscope, will be used to directly quantify the characteristics of observable porosity. At the same time, water absorption and density tests will be used to evaluate volume of porosity and the connectivity of the pores (ASTM C642¹, ASTM C1585)². Besides, the microstructure of samples will be investigated through Scanning Electron Microscopy (SEM). The hydration kinetics and type of hydration products present in the pastes will be estimated by Isothermal Calorimeter test (IC), Thermogravimetric analysis (TGA) and X-ray diffraction (XRD) analysis.

Tasks 4. Evaluation of transport properties. The bulk electrical resistivity and formation factor of the concrete samples will be estimated as per ASTM 1876-19. The Rapid Chloride permeability test (ASTM C1202³) will be used to evaluate the resistance of the concretes to chloride ions ingress. The transport properties will be assessed through the analysis of the results of this section in combination with the results from the water absorption test performed in task 3.

Tasks 5. Compressive strength of mortars and concretes. Compressive strength tests will be performed for each mixture design at 3, 7, and 28 days, according to ASTM C39⁴.

Task 6. Analysis of the results. A comparative analysis of the test results from tasks 1 to 5 for samples with and without nanoparticles and with and without CO₂ curing will be carried out. Then, the interconnection of the results of the different tests will be analyzed to acquire a deeper understanding of the combined effect of nanoparticle addition and CO₂ curing. This task will help with understanding the synergistic effects of CO₂ curing and nanomodification on the concrete's properties, providing insights into the optimal combination of parameters associated with these modifications.

Task 7. Draft of the report, Review and submission of Final report. This task will consist of the preparation, revision, and submission of the final report of the project, summarizing the research findings, methodologies, conclusions and recommendations.

Project Progress:

3. Progress for each research task

Task 1. Characterization of the Materials [100% completed]. Reported in the previous quarterly progress report.

Task 2. Preparation of specimens and curing process [80% completed] Pastes and mortars were mixed and prepared per ASTM C305⁶. Slump and spread of mortars were measured and collected per ASTM C1810⁷. Concretes were mixed per ASTM C192⁸, and their slump was measured per ASTM C143⁹.

After mixing and casting, the mortar and paste samples were cured under a tarp in the molds for 12 hours. Samples were then transported to an environmental chamber at 50%RH and left to cure for an additional 12 hours. Next, half of the samples were placed in a VWR Symphony CO₂ curing chamber at 60°C with an RH ranging from 60%-90%±5% and CO₂ concentration of 20% for 12 hrs. The other half of samples were placed in the environmental chamber with an RH of 50%±5% for 12 hrs. After this 12-hour period, all samples were cured in open-air at room temperature and approximately 40%RH after the comparison stage until their corresponding tests. A diagram of this process is provided in Figure 1.

CURING PROCEDURE : PASTES AND MORTARS

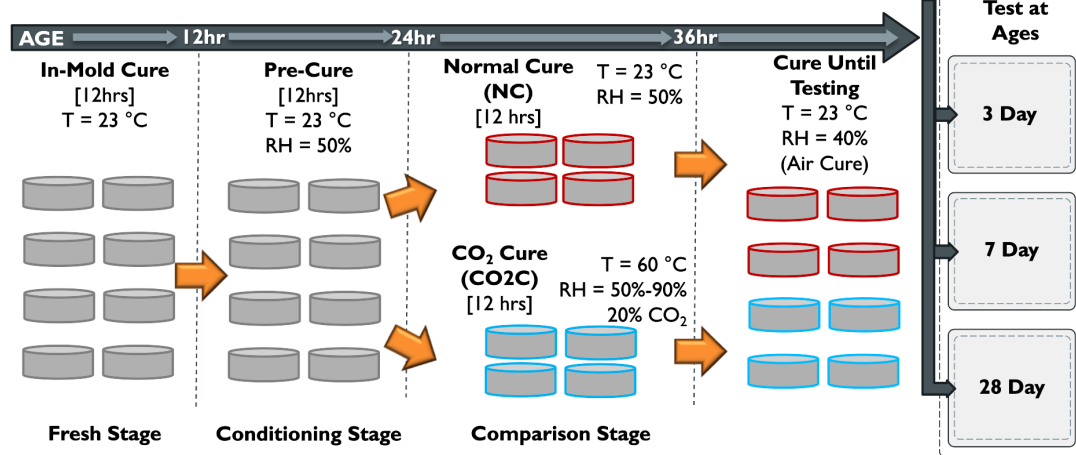


Figure 1. Paste and Mortar Curing Method over the First 36 Hours of Hydration

The curing procedure for concretes was adjusted to better imitate common precast curing methods. After mixing and casting, the concrete samples were cured in covered plastic molds for 12 hours. Half of the samples were then transported to an environmental chamber at $50\% \text{RH}$ and left to cure for an additional 24 hours, while the other half was submerged in a container of water at 50°C to imitate steam curing for 12 hours. Following this 12-hour period, the samples were placed into a TR-HTX CO_2 chamber at 50°C with an RH ranging from $70\% \pm 5\%$ and CO_2 concentration of 20% for 12 hrs. At 36 hours of age, all samples were cured in open-air at room temperature and approximately $40\% \text{RH}$ after the comparison stage until their corresponding tests. A diagram of this process is provided in Figure 2.

CURING PROCEDURE : CONCRETE

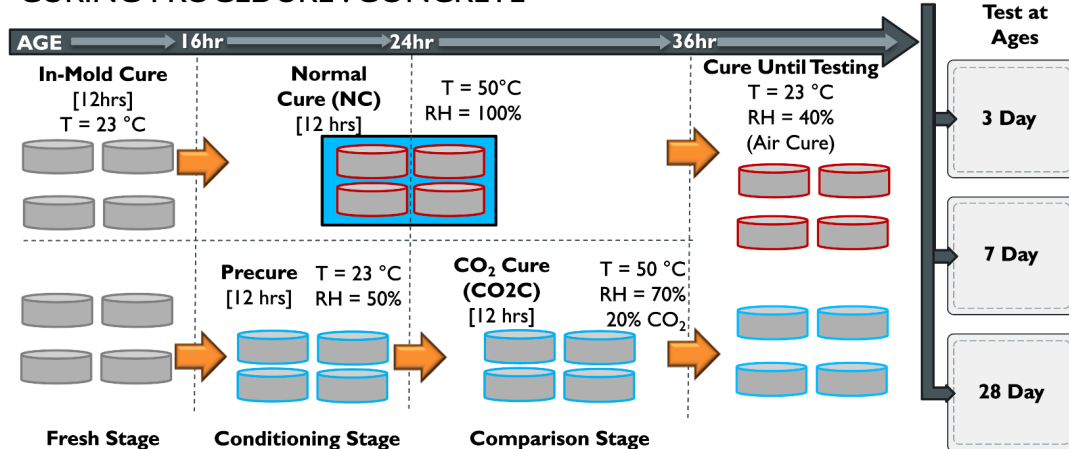


Figure 2. Concrete Curing Method over the First 36 Hours of Hydration

All the planned cement paste and mortar mixtures at 0.42 w/c ratios and 0.48 w/c ratios were mixed and cured. Reference concrete mixes and concretes with NS concentrations of 2% at 0.42 w/c ratio were mixed.

Task 3. Analysis of hydration process, porosity and microstructure [57% completed] Paste and mortar samples were prepared and epoxied for scanning electron microscopy (SEM). Reference concrete samples at 0.42 w/c ratios were mixed, cured, and prepped for absorption testing per ASTM C1585.

- Thermogravimetric Analysis (TGA) of Pastes. TGA was performed on pastes with a 0.48 w/c ratio and 0.42 w/c ratio at 3- and 7-day ages. The amount of $\text{Ca}(\text{OH})_2$ (CH) and calcium carbonate (CC) was quantified as per the *Kim and Olek method*¹⁰ for each mix. A 0.1-inch slice was cut from the surface of each paste sample, crushed, and ground to a fine powder for all TGA experiments. Isothermal calorimetry was performed for pastes with a 0.42 w/c ratio.
- Isothermal Calorimetry of Pastes. A TAM Air calorimeter was used in conjunction with ASTM C1702¹¹ to perform IC of pastes at 0.42 water-to-cement ratio. The IC test was performed for 7 days at room temperature (23°C). From the heat flow curves, total heat of hydration and thermal setting times were determined for each mix.

Task 4. Evaluation of transport properties [20% completed] Reference concrete samples were mixed, cured and prepared for rapid chloride ion penetration and resistivity testing per ASTM C1202 and ASTM C1876, respectively. To assess the concrete absorption, ASTM C1202 was selected for analysis of concrete properties instead of ASTM C642, as ASTM C1202 can provide more complete information regarding absorption than ASTM C642. The testing for the project mixtures will be completed once the corresponding curing time is reached.

Task 5. Compressive strength of mortars and concretes [70% completed]

- Compressive Strength of Mortars. Using ASTM C109, compressive tests were performed on mortars at 0.48 w/c and 0.42 w/c ratios at 3-day, 7-day, and 28-day ages. The average compressive strength of mortar specimens was then calculated.
- Compressive Strength of Concretes. Using ASTM C39, compressive tests were performed on reference concretes of 0.42 w/c ratios at 3-day ages. The average compressive strength of mortar specimens was then calculated.

Task 6. Analysis of the results [70% Completed] Notable differences between the composition and mechanical properties of CO_2 -cured and standard-cured cementitious specimens with and without nano-additives (nano-silica and recovered carbon black) at two different water-to-cement (w/c) ratios were observed. Thermogravimetric analysis shows that CO_2 curing greatly improved the CaCO_3 content in samples compared to standard curing. The incorporation of nano-additives is shown to increase the CaCO_3 content in higher w/c samples, especially when used with CO_2 curing. Compressive test results showed a strength increase in CO_2 -cured specimens compared to standard-cured specimens. Results indicated that the combination of both CO_2 curing and nano-additives usage resulted in the highest early compressive strength among all mixtures and curing conditions studied (which is very important for the precast production) while reducing the calcium hydroxide content, thus potentially reducing the risk of durability issues such as calcium oxychloride formation. Thus, results indicate that this combination can enable accelerated precast production while reducing the risk of durability issues of calcium oxychloride formation. Recovered carbon black, currently a waste product, may not only prolong the durability of mortars by improving strength but may also reduce costs and greenhouse gas emissions through its reuse in precast elements.

Task 7. Draft of the report, Review and submission of Final report. [50% Completed]. The information from the quarterly reports, plus additional detailed data, are currently included in the ongoing draft of the final report.

4. Percent of research project completed.

Approximately 75% of the total project has been completed to date.

5. Expected progress for next quarter.

By the end of next quarter, December 31st of 2024, 100% of this project (Phase I) is expected to be completed.

The remaining concrete specimens required to complete the experimental campaign will be cast and cured using the outlined concrete curing procedure by the end of October (Fig. 2). In addition, by the end of October the 28-day density, absorption, and resistivity data will be collected for all concrete samples. Within 65 days, rapid chloride permeability and pore volume will be complete for all concrete samples. Compressive strengths of all concrete specimens at 3 days, 7 days, and 28 days will be complete by the end of November.

Within the next 45 days, IC will be performed on mortar samples, and TGA will be performed on the remaining 28-day pastes. SEM will be complete within the next two months for 28-day pastes. XRD will be performed on all paste samples by December 25th, and the data will be incorporated into the draft of the final report.

All acquired data will be analyzed and formatted into a document by December 31st in preparation for the final report. The acquired data will be organized, analyzed, and submitted for publication within the next months.

6. Educational outreach and workforce development

The educational outreach and workforce development plan was outlined in the previous quarterly progress report. Future educational outreach and workforce development plans include the following:

Minority in Engineering Program's "Purdue Promise"

Purdue University's Minority in Engineering Program hosts "Purdue Promise" annually to encourage high school application and enrollment to Purdue university engineering schools. The Lyles School of Civil and Construction Engineering will host a tour for visiting highschoolers on October 13th to introduce students to fundamental engineering concepts and give them a preview of the innovative engineering technologies. This project will be featured with many others as a talking point for the tours.

Minority in Engineering Program's "Purdue Promise": Civil Engineering Tours.
Purdue University West Lafayette, Indiana
October 13th, 2024

Civil Engineering for a Day Correspondence (Featuring the current UTC-funded Project)

In Fall 2024, an engineering research event will be held at select High Schools offered by the graduate student working on the project. Conversations between high school administrators and the student have started, and dates have been scheduled. Select high schools are listed below:

Civil Engineering for a Day: Merrillville High School
Merrillville, Indiana
December 2024

Civil Engineering for a Day: West Side Leadership Academy
Gary, Indiana
December 2024

Civil Engineering Materials Tutorial Videos

In September and October, the graduate student will start recording videos showing how some selected experiments used during the project are conducted. These videos will be used to educate undergraduate students and introduce them to engineering principles, and to showcase our working procedures to prospective students interested in joining this project or others. These videos have a dual purpose in terms of workforce development: (i) to train the graduate student in terms of practicing and improving communication and teaching skills, and (ii) to create material that can be used for educational outreach activities that can connect easier with underrepresented groups, since the graduate student will be a role model for the next generation of researchers.

7. Technology Transfer

There is no progress on technology transfer yet.

Research Contribution:

8. Papers that include TRANS-IPIC UTC in the acknowledgments section:

No papers were published during this quarter. A draft of a research paper is in progress, and we expect to submit it by the end of the year. It is expected that out of the data acquired during this first year, at least two peer-reviewed research papers will be published in top journals of our field.

9. Presentations and Posters of TRANS-IPIC funded research:

- *TRANS-IPIC UTC Spring Workshop 2024 – April 22nd, 2024.*
The Grainger College of Engineering. (2024). 2024 Trans-IPIC UTC Workshop. 2024 TRANS-IPIC UTC Workshop. <https://trans-ipic.illinois.edu/workshop> (Fig. 3)
- *TRANS-IPIC UTC Monthly Research Webinar – June 17th, 2024.*
Trans-IPIC Monthly Research Webinar - June, 2024. Illinois Media Space. (n.d.). https://mediaspace.illinois.edu/media/t/1_xt7op9zl
- *United States Department of Transportation Future of Transportation Summit – August 13th, 2024. List of Posters. FoT Summit. (n.d.). https://fot-summit.org/?page_id=692 (Fig. 4).*

10. Please list any other events or activities that highlights the work of TRANS-IPIC occurring at your university. Similarly, please list any references to TRANS-IPIC in the news or interviews from your research.

The educational outreach activities are scheduled for October 13th and December 2024.

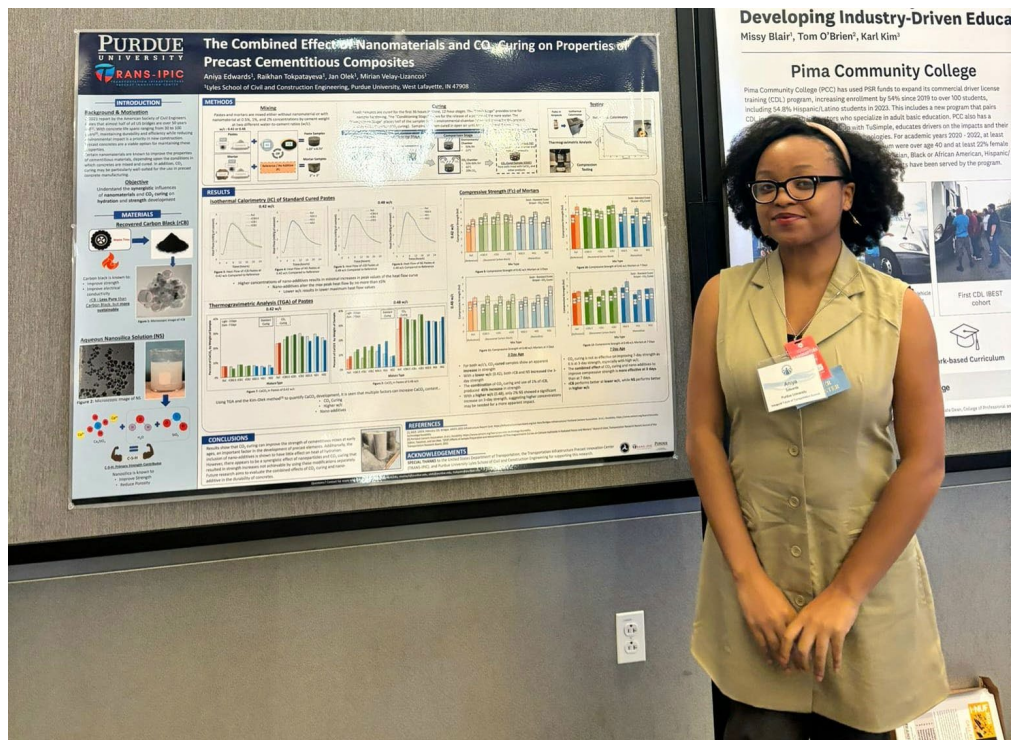


Figure 3. Poster presentation by the graduate student Aniya Edwards presenting the project results at the Future of Transportation Summit hosted by the USDOT at their headquarters in Washington D.C. in August 2024.

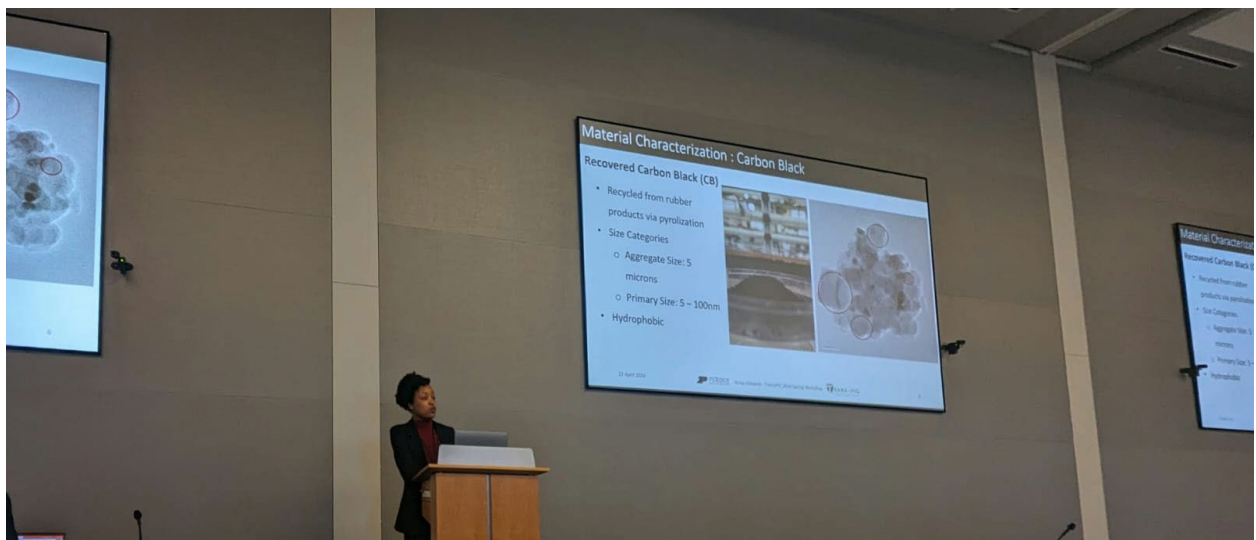


Figure 4. Research presentation by, Aniya Edwards (graduate student on this project), presenting the project results at TRANS-IPIC UTC Spring Workshop 2024, Chicago.

References:

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2. ASTM. C1585-20 Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic Cement Concretes. *ASTM Int.* Published online 2020:1-6. doi:10.1520/C1585-20.2
3. ASTM. C1202-22e1 Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration. *ASTM*. Published online 2022:1-8. doi:10.1520/C1202-22E01.2
4. ASTM. C39/39M-21 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens. *ASTM Int.* Published online 2021:1-5. doi:10.1520/C0039
5. ASTM E3294 - 23. Standard Guide for Forensic Analysis of geological materials by powder X-ray diffraction. *ASTM Int.* Accessed December 30, 2023. <https://www.astm.org/e3294-22.html>.
6. ASTM C305-20 Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency. *ASTM Int.* Accessed July 1, 2024. www.astm.org/c0305-20.html.
7. ASTM C1810/C1810M-23. Standard guide for comparing performance of concrete-making materials using mortar mixtures. *ASTM Int.* Accessed March 31, 2024. <https://www.astm.org/Standards/C1810.htm>
8. ASTM C192/C192M-19 Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory. *ASTM Int.* Accessed September 30, 2024 https://compass.astm.org/document/?contentCode=ASTM%7CC0192_C0192M-19%7Cen-US
9. ASTM C143/C143M-20 Standard Test Method for Slump of Hydraulic-Cement Concrete. *ASTM Int.* Accessed September 30, 2024 https://compass.astm.org/document/?contentCode=ASTM%7CC0143_C0143M-20%7Cen-US
10. T. Kim, J. Olek, "Effects of Sample Preparation and Interpretation of Thermogravimetric Curves on Calcium Hydroxide in Hydrated Pastes and Mortars", *Transp. Res. Rec. J. Transp. Res. Board*, 10-18, 2290 (2012).
11. ASTM C1702. Standard test method for measurement of heat of hydration of hydraulic cementitious materials using isothermal conduction calorimetry. *ASTM Int.* Accessed March 31, 2024. <https://www.astm.org/c1702-17.html>