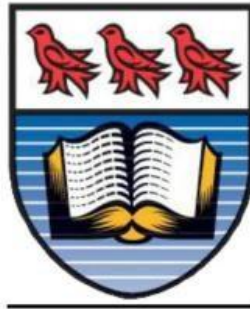


## **CIVE 693 – Candidacy Exam Proposal**

# ***CO<sub>2</sub> INJECTED 3D CONCRETE PRINTING: MULTISCALE CHARACTERIZATION***



UNIVERSITY OF VICTORIA

By  
Chirag Vijay Kumar

Supervisors:  
Dr. Rishi Gupta  
Dr. Bosco Yu

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## Introduction & Background

Concrete is everywhere—roads, buildings, bridges—but making it releases a lot of carbon dioxide ( $\text{CO}_2$ ). Reviews of the cement and concrete sector estimate that cement production alone contributes on the order of eight percent of global  $\text{CO}_2$  emissions, which is why researchers are exploring ways to turn concrete from a source of emissions into part of the solution. One promising idea is to store  $\text{CO}_2$  inside the concrete itself while it is still fresh. Under the right  $\text{CO}_2$  level, humidity, and exposure time, early-age carbonation turns some of the cement's hydration products into stable calcium carbonate minerals. This typically tightens the pore structure, speeds up early hardening, and reduces how easily water and salts move through the material, while locking carbon in place. In plain terms: we let the fresh material “breathe in” a controlled dose of  $\text{CO}_2$  so it mineralizes and becomes denser. (Chen et al., 2023; Kazemian & Shafei, 2023; G. H. Kim & Yang, 2025; Meesaraganda & Kazmi, 2025; Misyura & Donskoy, 2020; Monkman et al., 2016a; Possan et al., 2016; Sodeifian et al., 2025; W. C. Wang et al., 2024; Zhu et al., 2024)

3D concrete printing (3DCP) makes this approach especially attractive. A printer lays down soft “beads” of concrete in layers, creating many fresh surfaces and interfaces between layers. Those interfaces often act as the weak link; printed versions of the same mix can be 12–22% lower in compressive strength than cast specimens because of interlayer weaknesses. Improving the bond between layers—and doing so without harming the fresh beads—is therefore central to reliable printed parts. (L. Li et al., 2023a; Tay et al., 2023)

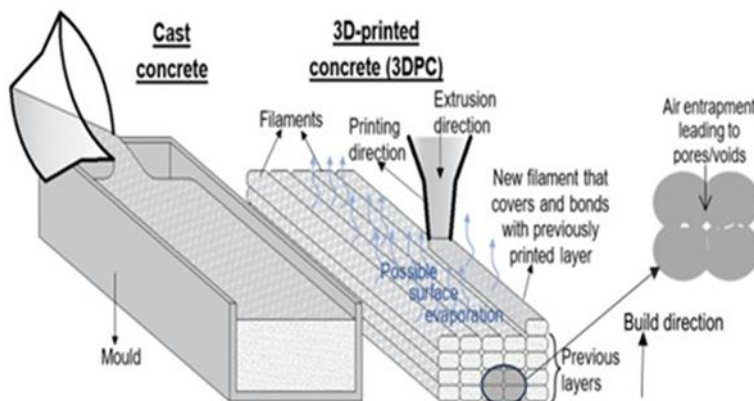


Figure 1: Picture showing cast concreting and 3D Printing

Workability vs. buildability. In traditional, cast-in-place concrete, accelerated carbonation can cut “open time” and reduce slump, making placement and finishing harder; in other words, early stiffening can be a workability penalty for ready-mix workflows. By contrast, 3DCP benefits from controlled early stiffening: higher early stiffness and yield stress improve shape retention and layer stability when they are timed to the layer cadence. The literature consistently reports early strength gains and lower absorption/chloride transport when

exposure is well managed, but also warns that over-carbonation or poor environmental control can embrittle surfaces or weaken layer interfaces—exactly what we aim to avoid in printing (Driver et al., 2024; Han et al., 2025; Kmentová et al., 2025; Lee et al., 2025; Pivezhani et al., 2016; Qu et al., 2025; D. Wang et al., 2023a, 2023b; Zajac et al., 2022). Several implementation pathways show the trade-offs. In post-printing chamber curing, exposing freshly printed parts to controlled  $\text{CO}_2$  levels tightened pore structure and improved interlayer bonding in mixes with recycled constituents; for example,  $\text{CO}_2$  curing reduced plastic shrinkage and increased both compressive and interlayer shear strength in printed mortars with recycled fine aggregate. Direct in-mix injection shows early-age strength benefits at modest doses by forming micro-scale calcium carbonate in paste voids. Jetting  $\text{CO}_2$  at the filament during deposition can raise uptake but risks disturbing the bead and reducing layer bond unless moisture and temperature are carefully controlled. (*Exploring Carbon Sequestration Potential through 3D Concrete Printing (1)*, n.d.; Seifu et al., 2023; Tay et al., 2023)

This proposal takes a practical, test-what-you-claim approach. First, we will define safe operating windows for  $\text{CO}_2$  dose and contact time that avoid overheating or surface damage. Next, we will test whether those settings measurably improve print quality (stable bead shape and stack height), interlayer bond, and early stiffness. Finally, we will scale to a small, printed element and count carbon fairly: stored  $\text{CO}_2$  is only credited if the element also passes basic performance checks (geometry, bond, and a quick durability). In short, our aim is to turn a high-emitting material into a verified carbon store—and to show, with measurements rather than assumptions, where the carbon goes, how much stays, and whether the

printed element performs like good concrete should. (Jiang et al., 2025; Kaliyavaradhan et al., 2020; Karulf et al., 2025; L. Li et al., 2023b, 2023a, 2023c; Wen et al., 2021; Zhong et al., 2024)

## Literature Review

The literature on CO<sub>2</sub> utilization in cementitious systems is broad and inconsistent in how exposure, humidity and timing are collected. Table 1 compiles the routes most relevant to 3D concrete printing (3DCP) system level mineralization, introduction system, early age curing, reactive surface treatments, RCA carbonation, and SCM-rich binders. So, we can compare typical settings, what they achieve, and where they fail. The right-hand column is intentionally pragmatic: it states why each route matters for 3DCP, i.e., whether it helps printability (shape retention, stack height), interlayer bond, or durability. Read the table as a design space rather than a scoreboard; the next sections extract the gaps this comparison reveals and translate them into research questions that drive the proposed work.

**Table 1. Evidence for CO<sub>2</sub> utilization routes relevant to 3D concrete printing (3DCP)**

Focus	Sources	Typical CO <sub>2</sub> setting / uptake	Observations	Key risks & controls	Why this matters for 3DCP
System-level accelerated carbonation & mineralization	(Kashef-Haghighi & Ghoshal, 2013; Monkman et al., 2016b; Zhu et al., 2024)	Broad bands reported: 5–150 kg/m <sup>3</sup> or 5–25% of binder, depending on method and mix.	Early-age strength ↑ (often +10–30%), pore refinement, permeability ↓, chloride transport ↓ when RH and CO <sub>2</sub> are controlled.	Over-carbonation → microcracks/shallow fronts; standardize dose–time–RH/T and QA logs.	Establishes credible result bands and the need for schedule-aware exposure and verification.
Ready-mix / inline direct injection (CRC)	(N. Kim et al., 2023; Possan et al., 2016; Zhang, 2024)	Injection within ~15–30 min of batching; ~8–15 kg CO <sub>2</sub> /m <sup>3</sup> stored (field-representative).	Early strength +10–20%; ITZ densification; sometimes lower water demand.	Plant timing & admixture interactions; enforce mixer/inline QA.	Translates to site logistics; informs same-schedule LCA assumptions.
Early-age chamber curing (post-print)	(X. S. Li et al., 2023; Quah et al., 2023; Seifu et al., 2023; D. Wang et al., 2023b)	~10–15% CO <sub>2</sub> , ~60–70% RH, 12–36 h typical.	ITZ tightening; permeability/absorption ↓; early strength ↑.	Over-exposure → surface hardening or pore blocking; control RH/T.	Clean fit for 3DCP via tent/chamber over print envelope; improves buildability and interlayer bond.
Reactive surface carbonation (thin coatings)	(Hao et al., 2022; Lee et al., 2025; Misyura & Donskoy, 2020; Peng et al., 2023; B. Sun et al., 2022; Xiong et al., 2024)	Minutes-scale conversion of reactive film.	Capillary absorption ↓; pull-off bond ↑.	Specify film thickness, RH, cure time.	Fast protective finish for printed façades / marine exposures.
Recycled concrete aggregate (RCA) accelerated carbonation	(Sousanabadi Farahani et al., 2025; Wu et al., 2025; Zou et al., 2021)	Uptake in residual paste of RCA; depends on moisture/size class.	Concrete with carbonated RCA: strength +10–25%; absorption –20–40%.	“Shell-only” carbonation if moisture control is poor.	Stiffer, less-absorptive mixes stabilize extrusion rheology and interlayer bonding without excess paste.
Ternary SCMs (metakaolin + limestone)	(Schöler et al., 2017)	Reported binder-mass uptake bands; carboaluminate formation.	Early strength ↑; sulfate resistance ↑ via carboaluminates.	Tune MK/LS fractions, CO <sub>2</sub> %, RH to avoid decalcification.	Low-clinker route that responds positively to controlled CO <sub>2</sub> for printable elements.
High-slag binders (GGBFS)	(Alonso-Cañón et al., 2025; Ashraf & Olek, 2018; Baghi et al., 2024; Kaliyavaradhan et al., 2020)	Moderate %CO <sub>2</sub> ; hours-to-days exposure windows.	~+30% early strength; pore mode shifts to 10–50 nm.	Over-exposure (>7 d) risks decalcification.	Fast-turnaround precast/printed modules without steam.

Focus	Sources	Typical CO <sub>2</sub> setting / uptake	Observations	Key risks & controls	Why this matters for 3DCP
3DCP with recycled fines (post-print CO <sub>2</sub> )	(Ding et al., 2020; Lee et al., 2025; B. Sun et al., 2022)	Gentle chamber curing after deposition.	Plastic shrinkage ↓; compressive & interlayer shear ↑.	Avoid filament disturbance; control RH/T.	Direct evidence that post-print CO <sub>2</sub> can stabilize layers and boost bond.
UHPC under CO <sub>2</sub>	(Briki et al., 2021; Kaliyavaradhan et al., 2020; Kmentová et al., 2025; Q. Liu et al., 2024; Schöler et al., 2017; Wen et al., 2021; Zajac et al., 2022)	Shallow fronts in very dense matrices.	Small early compressive ↑; near-surface pores refined.	Limited storage (depth-limited).	Use for surface-level upgrades; not a storage workhorse.
Alkali-activated materials (AAMs)	(Amran et al., 2022; Bawab et al., 2025; J. Sun et al., 2025)	Chemistry-dependent; prefer Ca-rich systems.	Ca-rich: permeability ↓ with acceptable strength; low-Ca FA: risk of gel depolymerization/pH drop.	Keep mild CO <sub>2</sub> and conservative windows; verify before use.	Targeted use only; protect long-term alkalinity & steel passivation.

*Abbreviations: RH = relative humidity; ITZ = interfacial transition zone; FA = fly ash; MK = metakaolin; LS = limestone powder; UHPC = ultra-high-performance concrete; CRC = carbonation-ready concrete.*

Table 1 puts together the main CO<sub>2</sub>-utilization routes (system-level mineralization, ready-mix/inline injection, early-age chamber curing, reactive surface coatings, RCA carbonation, SCM-rich binders, UHPC, and AAMs) and distills what they typically achieve, where they can fail, and why they fit 3D concrete printing (3DCP). Across the board, controlled carbonation converts part of the cement's hydration products especially calcium hydroxide and portions of C-S-H into stable calcium carbonate, which tightens pores and can increase early stiffness/strength when humidity, CO<sub>2</sub> level, and time are set correctly. Typical ranges and what they mean: As mixes and test methods differ, reported CO<sub>2</sub> uptake spans single-digit to >100 kg/m<sup>3</sup> and early-age strength gains are often ~10–30% under ~10–20% CO<sub>2</sub> and ~60–70% RH bands echoed across reviews and experimental studies cited in the table. These improvements track with pore-size refinement (toward ~10–50 nm) and lower sorptivity/chloride transport signals linked to better durability. **Why 3DCP especially benefits:** For cast-in-place concrete, accelerated carbonation can shorten open time and reduce slump, which hurts workability. In 3DCP, the same controlled early stiffening is an asset: it improves shape retention and layer stability when timed to the layer cadence, and it can strengthen the interlayer bond—the usual weak point in printed parts. The post-print chamber pathway in Table 1 aligns naturally with printing (tent/chamber over the print envelope), while inline/ready-mix dosing informs batch logistics and same-schedule LCA.

### Identification of Research Gaps from Table 1:

- **CO<sub>2</sub> Introduction Window:** System-level mineralization gives the broadest result bands (uptake and early strength) but also the widest variability hence the need to regulate correct dose–time–RH/temperature and QA logs when moving from lab to practice.
- **Admixture interaction uncharacterised:** Ready-mix/inline injection shows ~8–15 kg/m<sup>3</sup> stored and +10–20% early strength when metered ~15–30 min after batching; plant timing and admixture interactions are the key controls.
- **Interlayer-Bond under measured:** Early-age chamber curing (~10–15% CO<sub>2</sub>; ~60–70% RH; 12–36 h) tightens the ITZ and reduces permeability—useful for buildability and interlayer bond in 3DCP.
- **Reactive coating and carbonated recycled concrete aggregate:** Reactive coatings carbonate within minutes and lower capillary absorption while raising pull-off bond—a fast finish for façades or marine exposure. RCA carbonation upgrades aggregate (less absorption, harder surface) and improves concrete strength by ~10–25% when moisture/size are controlled; for 3DCP, this stabilizes extrusion rheology and interlayer bonding without excess paste.
- **Supplementary Cementitious Materials (SCMs) not tuned to printing:** SCM-rich binders (slag-rich; MK + limestone ternaries) show synergy under controlled CO<sub>2</sub>: ~30% 3-day strength in

high-slag systems and carboaluminate formation in MK+LS, with pore-mode shifts into the fine range good news for low-clinker printable binders.

- **Dense mixes store CO<sub>2</sub> only near the surface:** UHPC benefits are surface-limited (dense matrix), so use it as a surface upgrade, not as a storage workhorse.
- **Alkali-Activated Materials (AAMs):** AAMs are chemistry-dependent: Ca-rich (slag) can gain; low-Ca/high-FA can suffer gel depolymerization and pH drop if pushed so keep mild CO<sub>2</sub> and conservative windows, verified before adoption.
- **Element-Level Proof of Performance:** Most work stops at paste/mortar scale. We will test a printed element and grant any CO<sub>2</sub> credit only if all performance criteria are acceptable.

### Risks and why controls matter: -

Based on the literature review, currently every methodology that has been conducted as led to failure when exposure to CO<sub>2</sub> exceeds a safe window. Too much CO<sub>2</sub> causes micro cracks, shallow fronts and blocked pores that reduce durability and layer bond. We must limit the temperature to a certain acceptable limit and so do these also RH and log every trials.

### Mechanisms and pathways background for Table 1

1. Chemically, accelerated carbonation/mineralization targets portlandite and parts of C-S-H to form CaCO<sub>3</sub>, densifying the microstructure and typically increasing early stiffness/strength when RH (~60–70%), CO<sub>2</sub> (~10–20%), and time are tuned to the mixture. Reported uptake ranges run from single digit to >100 kg/m<sup>3</sup>, explaining the spread seen in Table 1's cells.
2. Process routes for 3DCP mirror Table 1: (i) direct injection to fresh mix/line (~15–30 min window; ~8–15 kg/m<sup>3</sup> stored), (ii) early-age chamber after deposition (~10–15% CO<sub>2</sub>; 12–36 h), and (iii) reactive coatings as a rapid finish. Each enhances different parts of the system batch logistics, interlayer zone, or surface durability.
3. Performance and durability: Studies report ~15–30% early-age strength gains (24–48 h), pore refinement to ~10–50 nm, and measurable drops in sorptivity and chloride migration with the bonus that carbonation depth in dense matrices is usually shallow, preserving core hydration.
4. SCM synergy and circular materials: Fly ash, high-slag, and MK+LS ternaries respond well under controlled CO<sub>2</sub>, delivering faster early gains and better transport resistance; RCA carbonation upgrades aggregate to help buildability in prints.
5. AAMs: Useful but not universal Ca-rich systems tolerate moderate CO<sub>2</sub>; low-Ca mixes need caution to protect long-term alkalinity and steel passivation.
6. Standards and verification: The literature repeatedly calls for harmonized uptake methods (mass-gain/TGA), depth, and permeability benchmarks, plus clear code pathways exactly the kind of audit trail your study proposes to provide.

### Summary of gaps and novelty of proposed study

1. Process control vs. real printing. Most studies ran chambers or small molds without tying dose/contact to layer cadence/open time/stack height; **Novelty: synchronize exposure to printing.**
2. Interlayer bond is under-measured. Many papers report compressive strength/durability but lack direct tension or slant-shear across printed interfaces; **Novelty: elevate interlayer bond to a primary metric.**
3. Geometry and stability rarely gated. Few enforce “did it print correctly?” checks (±10% bead/wall, stable stack); **Novelty: add printability acceptance criteria.**
4. Thermal/RH not actively managed. Exposure windows often ignored temperature/moisture limits; **Novelty: use IR + RH control and abort criteria.**
5. Uptake claims without performance proof. Some credit stored CO<sub>2</sub> without verifying structural/durability targets; **Novelty: adopt “no pass → no credit” in LCA.**
6. Lab results not promoted to elements. Many stops at pastes/mortars; **Novelty: test a small, printed element and only count carbon for a passing element.**
7. Standards/audit trail missing. Reporting of methods/logs is inconsistent; **Novelty: build a full audit trail (schedule, geometry, properties, carbon) mapped to referenced test methods.**

### Research Questions

- **RQ1 — Carbon-dioxide setting.** What dose (as a percentage of binder mass) and what contact timing produce early mineralization without harmful temperature rise or surface chalking?

- RQ2 — Printability and interlayer bond. Under those settings, can bead and wall geometry remain within tolerance, stacks remain stable, and layers bond strongly?
- RQ3 — Structural checks. When a small, printed element is made with the same settings, does it meet load and service criteria without failure along a weak layer?
- RQ4 — Carbon impact under the same schedule. Does the net global-warming potential decrease when carbon credit is granted only after all acceptance criteria are passed?

### Expected contributions (what this study adds)

- Method → not numbers- A repeatable method to find the right carbon-dioxide setting for *any* mix: small pilot matrix (dose × contact time), temperature safety cap, and go/no-go rules. You tune it locally; the playbook stays the same.
- Printability envelope procedure - A simple way to map “can it print and hold shape?” using bead/wall checks now and at fifteen–thirty minutes, plus a stable-stack test. Outputs are pass/fail against a tolerance you set project-by-project.
- Interlayer bond protocol (normalized) -Pull-off and slant-shear across layers, always reported relative to the same-day control. Because it is normalized, the data travel across mixes and plants.
- Pass-gated carbon accounting - A credit-only-when-it-passes rule and a logging template (materials, energy, transport, waste, measured storage). Works with any supplier and any binder—no fixed recipe assumed.
- Acceptance checklist you can drop into specs - Plain language criteria (geometry within tolerance, stack height achieved, bond  $\geq$  control, quick durability improved) that owners can put in tenders—with the tolerance value left blank for local choice.
- Standard data schema and forms - Ready-to-use sheets for dose, timing, printer speed, surface temperature, ambient, materials, energy, transport, waste, and lab results—so teams can audit and compare across jobs.
- Two mix-independent indices – (1) Interlayer Bond Index (IBI): bond of printed coupon  $\div$  bond of same-day cast/control. (2) CO<sub>2</sub> Uptake Index (CUI): measured stored CO<sub>2</sub>  $\div$  binder mass (or  $\div$  element volume) for passing prints only. Both are dimensionless and comparable across mixes.
- Risk controls and stop rules - A short list of abort conditions (temperature-rise cap, surface chalking, geometry drift beyond tolerance) that any crew can apply without changing a mix.
- Training and commissioning flow - A three-step onboarding for new plants: (1) calibration matrix, (2) printability and bond gate, (3) one element and pass-gated carbon. This reduces trial-and-error regardless of the recipe.

## Research Proposal

### Objectives:

#### Micro — Material & Microstructure

- Map the pore structure with Scanning Electron Microscopy and quantify pore size distribution and total porosity.
- Link pore structure metrics to durability indicators such as absorption and chloride transport.
- Build a simple early-age strength model for zero to seventy-three hours using Ultrasonic Pulse Velocity and confirm mineral formation with spectroscopy and thermogravimetry.

#### Meso — Process & Interface

- Prove clean printing: stable bead and wall geometry and a stack that holds its shape.
- Measure the interlayer bond under direct tension and under slant-shear and compare against the control.
- Control temperature rise during mixing and printing to avoid surface chalking and thermal cracking.
- Document the full printing window: layer timing, contact time, and environmental conditions.

#### Macro — Structural Performance

- Test a printed element (panel or beam) for load and service behaviour and confirm failure is not along a weak layer.
- Evaluate long-life behaviour under small, repeated load cycles.

- Check response to lateral actions: low-cycle bending or seismic-type loading, and wind-induced comfort effects where relevant.
- Assess fire performance of printed elements with a simple thermal exposure and residual-strength check.
- Investigate reinforcing-bar bond in printed material with a pull-out test.
- Trial overlays and jacketing as retrofit strategies on printed substrates and measure interface strength.

## Experimental Methodology

### Overview

This study advances in three stages that move from material behaviour to process behaviour and finally to element-scale verification. The goal is to show that early-age, printing-aware exposure to carbon dioxide can permanently store carbon while preserving or improving printed quality. A common operating window is enforced across all stages: the carbon-dioxide dose is selected in three bands (low, mid, and high as a percentage of binder mass), contact is applied for defined durations beginning at the start of mixing or at clearly defined moments during deposition, and the environment is held within a controlled range of relative humidity and ambient temperature—ranges consistent with accelerated carbonation curing practice. An infrared camera continuously records surface temperature; any schedule is aborted if the temperature rise exceeds a pre-set cap or if a chalky skin appears. Replication is built in by testing multiple specimens of each type, and every schedule will be compared to two same-day controls: a cast control and a printable control with no carbon-dioxide exposure.

### Phase One: Micro (material and microstructure)

The objective is to **lock a printable base mixture** and to **identify safe, effective windows of dose and contact time** that increase early stiffness and form measurable calcium carbonate without surface damage. The mixture uses **a fixed water-to-binder ratio, a defined sand grading, and a standard combination of superplasticizer and viscosity modifier**. **Four batches are made: a control with no carbon-dioxide exposure and three levels with in-mix dry-ice dosing**. Early behaviour over the first six hours is tracked by **recording Ultrasonic Pulse Velocity at regular intervals and converting those readings into a relative stiffness scale**; a vane or needle check records the time at which the paste shows no visible slump. Infrared temperature is recorded throughout, and any overshoot above the temperature-rise cap triggers an immediate stop. To confirm mineral formation, **paste samples are tested by Attenuated Total Reflectance–Infrared Spectroscopy and by Thermogravimetric Analysis at approximately two hours and again at twenty-four hours**. To gauge whether transport pathways are tightening, **small prisms are tested using water absorption or a short-duration Rapid Chloride Permeability surrogate**. This “mechanism-plus-performance” approach follows established carbonation-curing protocols (Lu et al., 2024; Osman et al., 2021; Zajac et al., 2022; Zhu et al., 2024).

### Phase Two: Meso (process and interface)

The aim is to show that **the same windows of dose and timing produce clean printing and strong interfaces between layers**. A SCARA operating at a specified speed and nozzle diameter lays down straight beads and short walls for each schedule that passed the Micro stage. Bead width and height and wall height are measured immediately after placement and again after fifteen to thirty minutes to evaluate geometric stability during the open-time window; walls are then built to the maximum height that can be stably stacked, and any failure is logged as buckle, slide, or collapse. Interlayer bond is quantified on coupons cut across layer interfaces using direct-tension pull-off and slant-shear tests at defined angles; for each schedule, the mean value, the standard deviation, and the ratio to the printable control are reported. The environmental window is maintained, and the infrared camera continues to enforce the temperature cap. The selected measurements and acceptance logic are aligned with reports on post-print chamber curing and interface strengthening in printed mortars, including those with recycled fines. ***The Ultrasonic Pulse Velocity–based early-age strength model is used here to select layer-to-layer timing and to verify that printing windows chosen in practice align with the predicted strength and stiffness gain.***

Field validation using the SCARA Road Runner. **The same in-mix carbon-dioxide method will be verified on the SCARA printer**. Targets are bead width and wall height within ten percent of target; a full, time-stamped log of carbon-dioxide dose, contact timing, printer revolutions per minute, surface temperature, and ambient conditions. One baseline print with no carbon-dioxide



and two carbon-dioxide schedules will be run. Specimens include one single-pass line and one multi-layer wall; cut samples are returned for layer-bond and mineralization checks. The field sequence is print line → print wall → verify size and height (ten percent tolerance) → cut samples → export the complete log for the life-cycle assessment. (Chandra Kishen et al., n.d.; Curth et al., 2024; Davolio et al., 2025; Fetzer et al., 2025; Kashef-Haghighi & Ghoshal, 2013; Khan & Koç, 2023; Kristombu Baduge et al., 2021; Krstulovic & Dabic, n.d.; Lachmayer et al., n.d.; Quah et al., 2023; Rymeš et al., 2024; Tarhan & Şahin, n.d.; Wolfs & Suiker, 2019; Žitek Makoter et al., 2025)

### **Phase Three: Macro (element-level proof and fair carbon accounting)**

A single small element—a panel or beam printed with the same settings that passed the Meso stage—is tested with engineering methods to confirm structural adequacy and to compute net carbon fairly. The element is loaded in four-point bending, or in compression where appropriate, while load versus deflection is recorded and a crack map is kept; the failure mode is checked to ensure it is not governed by a weak layer. Specimens are cut from the printed element to repeat the same quick durability used earlier (absorption or short-duration RCPT). A fair carbon balance is then performed: net global-warming potential equals materials plus mixing, pumping, and printing energy plus transport, minus measured mineralized CO<sub>2</sub> obtained from spectroscopy and thermogravimetry on samples from the element. Carbon credit is reported only if the element meets the geometric, interlayer-bond, and durability criterias. This sequencing follows the literature emphasis on chamber control, deployment practice, and verification before crediting carbon storage . Life-cycle assessment scenarios. Results are reported for three scenarios: S0 (cast, no carbon-dioxide), S1 (cast, in-mix carbon-dioxide), and S2 (three-dimensional-printed, in-mix carbon-dioxide). For each scenario we compute Net global-warming potential = materials + mixing, pumping, and printing energy + transport – measured stored carbon-dioxide, and we show the percent change relative to S0 with a small uncertainty bar. Acceptance conditions for carbon credit. Carbon credit will be reported only when all of the following are satisfied by the printed element made with the same schedule: (1) bead width and wall height are within the defined geometry tolerance; (2) the wall stack reaches its target height without failure; (3) the interlayer bond is equal to or greater than the printable control; (4) water absorption is lower than the control; (5) the rapid chloride permeability (short-duration surrogate) is lower than the control; (6) the carbonation depth meets the project’s limit for the selected exposure class; and (7) Attenuated Total Reflectance–Infrared Spectroscopy shows a carbonate signal at or above the set threshold for acceptance. (Davolio et al., 2025; Jin et al., 2025; Kazemian & Shafei, 2023; N. Kim et al., 2023; B. Liu et al., 2021; Pan et al., 2023; J. Sun et al., 2021)

(Net global-warming potential = materials + mixing, pumping, and printing energy + transport and waste – measured stored carbon-dioxide.)

## **Analysis and Modelling of Results**

### **Interpreting the Micro stage:**

Ultrasonic Pulse Velocity time-series will be converted into relative stiffness curves, and the no-slump timestamp will be extracted from the vane or needle check. Infrared temperature logs will be reviewed to verify that all data being analysed stayed at or below the temperature-rise cap; runs that exceeded the cap are excluded. Spectroscopy and thermogravimetry traces will be interpreted to confirm the presence of calcium-carbonate peaks or mass-loss signatures associated with carbonate formation. Scanning Electron Microscopy images will be taken at consistent magnification and field of view to avoid edge effects, and simple counts of void frequency and average void area will describe pore refinement. The quick durability indicator—absorption or short-RCPT—will be expressed as a percent change relative to the same-day control and plotted against the pore metrics to demonstrate that fewer and smaller pores coincide with lower transport. Advancement will require a smooth, increasing stiffness curve, confirmed carbonate formation, reduced transport relative to the control, and no temperature-cap violations—signals synthesised across accelerated-carbonation reviews.

### **Interpreting the Meso stage**

Bead and wall measurements taken immediately after placement and again at fifteen to thirty minutes will be compared with their targets to quantify tolerance and to define a practical printability envelope that captures combinations of layer timing and bead stability that maintain geometry and permit stable stacking. Interlayer bond will be summarised as the mean and standard deviation for both pull-off and slant-shear, and each schedule will also be reported as a ratio to the printable control, so acceptance is

plain. The acceptance criteria are straightforward: geometry within plus or minus ten percent, stack height achieved without failure, and interlayer bond equal to or greater than the control with a preferred margin of ten percent; thermal safety will be documented by the peak temperature rise and the time spent at or above the cap. These outputs will also use to fit a two-parameter interlayer representation for the later parity check (tension intercept from pull-off; effective shear from slant-shear). Emphasis on geometric stability, stack ability, and interlayer performance will follow successful process- and interface-quality studies.

### **Interpreting the Macro stage.**

The element test will be first checked for sensor artefacts or warm-up effects. The measured peak load and the service-level deflection will be compared with the target values, and the observed failure mode will be cross-checked against the two-parameter interlayer representation derived from the Meso stage; parity will be acceptable when the predicted failure locus matches the experiment and the predicted and measured peaks differ by no more than a practical ten percent band. The durability from the element will be compared with the same-day control using the same “lower is better” rule. The net global-warming potential will then be computed from material quantities, measured energy for mixing, pumping, and printing, transport distances, and the measured mineralized CO<sub>2</sub> from spectroscopy and thermogravimetry. Ninety-five percent confidence intervals will be displayed on the key plots—interlayer bond, geometry tolerance, and net global-warming potential—to communicate uncertainty. The decision rule is unchanged: a schedule passes only when every gate passes; only then is the carbon credit reported—consistent with pass-gated accounting recommended in reviews and deployment papers. After the Stage Three element passes all acceptance criteria, a short set of exploratory checks will be performed without delaying the primary deliverables. These checks probe risks highlighted in practice—low-cycle behaviour, wind comfort, fire exposure, reinforcing-bar bond, and retrofit overlays—using small, fast tests. Results will be reported as exploratory only; carbon accounting remains tied to the Stage Three pass.

**Low-cycle loading (seismic):** A small, printed specimen made with the same schedule is subjected to controlled load–unload cycles at increasing amplitudes. We will record hysteresis loops, stiffness reduction per cycle, residual deflection, and a crack map. The check is considered satisfactory when loops are stable within the planned range and no premature interlayer slip governs the response. **Wind-comfort:** For thin panel elements we apply a gentle, repeated lateral excitation (or its quasistatic equivalent) to observe drift, vibration comfort indicators, and any onset of serviceability issues such as rattling at interfaces. The check is considered satisfactory when measured deflection and response remain within the target comfort band and no interlayer distress is observed.

**Thermal Exposure:** Coupons sawn from the printed element mid-height are exposed to a defined temperature profile in a ventilated oven and then cooled to laboratory conditions. We will measure residual pull-off strength or residual compressive strength and document visible damage. The check is considered satisfactory when residual strength remains within the project’s target band and there is no interlayer delamination on visual inspection. **Reinforcing-bar bond:** Short prisms or cylinders printed with the same schedule will be cast or printed around a reinforcing bar that crosses layer interfaces. A monotonic pull-out test measures peak bond stress, slip at peak, and failure mode. The check is considered satisfactory when peak bond meets the target relative to the cast control and failure is not governed by a weak interlayer at service-level stress.

**Retrofit overlay & jacketing:** A thin overlay or jacketing layer is applied to a printed wall segment. After the specified cure, pull-off tests (and where appropriate, slant-shear tests) are performed through the overlay into the printed substrate. The check is considered satisfactory when overlay interface strength meets or exceeds the baseline, and failure occurs away from a weak interlayer plane. All specimens will use the same schedule that passed Stage Three (same dose, timing, and environmental controls). Test logs will include the loading or thermal profile, cycle counts or exposure time, specimen geometry, and any deviations. These validations will be scoped to be short and informative; if timing or resources constrain the work, they will be deferred without impacting the main claim.

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## Appendix

- **Appendix A: Appendix A-Project Schedule**
- **Appendix B: PhD Thesis planner-Chirag Vijay Kumar**
- **Appendix C: Operating Windows & Test Plan**

**Common CO<sub>2</sub> Curing Parameters:** All experiments will use a controlled carbonation environment in line with accelerated curing practice. Target gas concentration is  $\approx 10\%$  CO<sub>2</sub> (by volume), with low ( $\approx 5\%$ ) and high ( $\approx 20\%$ ) dose bands to bracket effects. Relative humidity is maintained at 60–70% RH (target  $65\% \pm 5\%$ ) and ambient temperature at  $\sim 25^\circ\text{C}$  to mimic standard accelerated-curing conditions. An infrared thermal camera continuously monitors specimen surface temperature; the run is auto aborted if surface  $\Delta T$  exceeds  $\sim 5^\circ\text{C}$  above baseline (approximately  $>30^\circ\text{C}$  absolute) to prevent thermal cracking or “chalky” carbonation layers. CO<sub>2</sub> exposure durations are selected based on mix and scale: for in-mix carbonation, 30–180 seconds of sealed contact in the mixer (per batch); for chamber curing of printed parts, typical periods of  $\sim 12$ , 24, and 36 hours will be tested. These windows reflect practical limits where benefits plateau and over-carbonation risks (microcracking, pore blockage) must be avoided.

**Equipment & Nozzle Specifications:** A gantry-style 3D printer with a progressive cavity pump will extrude the mortar through a 20 mm diameter nozzle (square profile). This nozzle size is chosen per literature on printable mortars (sand  $< 2$  mm) to ensure consistent bead geometry. The printer enclosure will be shrouded with a clear plastic tent to serve as a curing chamber for post-print CO<sub>2</sub> exposure. Gas is introduced either by sublimating pre-weighed dry-ice pellets (solid CO<sub>2</sub>) or via a mass-flow-controlled inlet for gas, depending on stage: in Phase 1 (mix-level dosing), crushed dry ice is added directly into the mixer at the start of mixing (lid immediately secured); in Phase 2 (early-age curing), gaseous CO<sub>2</sub> is released into the tent around the fresh print. In all cases, electronic sensors log CO<sub>2</sub>%, temperature, and RH in real-time inside the chamber or mixer. A ppm CO<sub>2</sub> sensor with alarm is also placed in the lab to warn if any leak causes ambient levels  $>0.5\%$  (5000 ppm) for safety.

**Replication & Controls:** Each test condition will be run with at least 3 specimens ( $n = 3$ ) to ensure statistical reliability. Results will be reported as mean  $\pm$  standard deviation for properties like strength, sorption, etc. All CO<sub>2</sub>-treated batches have two reference counterparts cured concurrently: (1) a conventional cast control (no printing, no CO<sub>2</sub>) to benchmark material performance, and (2) a printed control (printed identically but with no CO<sub>2</sub> exposure) to isolate the effect of carbonation. Performance metrics must meet minimum acceptance thresholds: no CO<sub>2</sub>-treated sample should underperform the non-CO<sub>2</sub> printed control in any structural or durability metric. In practice, this means the printed CO<sub>2</sub> samples must match or exceed control compressive strength, have no interlayer bond reduction relative to non-CO<sub>2</sub> prints, and show equal or lower absorption/chloride penetration than controls. Any schedule that produces visible flaws (e.g. surface powdering, new cracks, layer delamination) or causes a drop in strength or durability below control will be flagged as unacceptable. Those parameters will be adjusted or eliminated in subsequent iterations. By enforcing these criteria – along with the real-time sensor cut-offs for temperature and humidity – the operating window for “safe and effective” CO<sub>2</sub> curing will be systematically narrowed down.

**Planned Measurements:** For each curing schedule, early-age properties (e.g. 1 day and 3 days compressive strength, 3-hour ultrasonic pulse velocity gain) will be measured to capture the immediate effects of CO<sub>2</sub>. Buildability metrics (e.g. maximum unsupported layer count, achieved layer height vs. target) will document print quality under each condition. Hardened performance at 28 days will include compressive strength, interlayer tensile bond strength, four-point flexural strength (for printed beams), and transport properties (24 h water absorption, rapid chloride migration coefficient). Results will be tabulated as mean  $\pm$  SD and compared against the control benchmarks. A successful CO<sub>2</sub> curing schedule is one that improves early stiffness/strength and/or durability without compromising print geometry or bond. As an additional acceptance gate, the project’s carbon accounting will only credit CO<sub>2</sub> uptake from schedules that meet or exceed all control performance – this ensures no sustainability trade-off at the expense of quality.

- **Appendix D: CO<sub>2</sub> & Dry-Ice SOP + Safety Controls**

**CO<sub>2</sub> Handling and Injection SOP:** All carbonation curing will follow a strict protocol to ensure consistency and safety. For mix-phase carbonation (Phase 1), the procedure is: pre-weigh the required dry-ice (solid CO<sub>2</sub>) dose in a vented container (wear cryogenic gloves and face shield), add it to the mixer immediately after water and before high-speed mixing, then clamp down the mixer lid with a foam gasket seal. The

dry ice will sublime in the sealed mixer, providing the CO<sub>2</sub> dose for ~2–3 minutes of mixing. After the set contact time (monitored by a stopwatch and logged), the mixer is opened in a fume hood or well-ventilated area to release any excess CO<sub>2</sub> before retrieval of the fresh mix. Purge the mixer with fresh air for >1 min before an operator looks in. For early-age chamber curing (Phase 2 and 3), once a specimen or printed section is placed in the curing tent (an airtight plastic enclosure over the print bed), CO<sub>2</sub> gas from a cylinder is introduced through a regulator and diffuser. Gas flow is initiated at a low rate to displace air until reaching the target 10% concentration – verified by an in-tent CO<sub>2</sub> sensor. The tent or chamber is then sealed for the designated curing period. Humidity is maintained by placing a damp sponge or ultrasonic humidifier inside to target ~65% RH (to prevent the sample from drying out). Throughout exposure, an IR camera tracks surface temperature (see below), and periodic readings from the CO<sub>2</sub> and RH sensors are recorded in the log. After curing, the chamber is vented slowly (unzipped or unsealed in partial openings) in a fume-hood or outdoor area to avoid a surge of CO<sub>2</sub> into the lab.

**Sensor Thresholds & Automation:** A multi-channel data-logger will continuously monitor: (a) CO<sub>2</sub> concentration inside the curing enclosure, (b) relative humidity and temperature, and (c) specimen surface temperature via IR thermography. Thresholds are programmed such that if CO<sub>2</sub> exceeds the setpoint by >1% (absolute) or RH deviates outside 60–75%, the mass flow controller or humidifier will auto-adjust to compensate (or shut off CO<sub>2</sub> if out-of-range). Crucially, if the IR camera detects the specimen surface exceeding 30 °C (the thermal “cap” determined to prevent damage), an alarm triggers and the CO<sub>2</sub> inlet is shut off automatically. In practice, a sustained temperature rise of ~5 °C above ambient is treated as the cutoff for safety. At that point, fans will purge the chamber/tent with fresh air to cool the sample. Similarly, the appearance of a “chalky” white surface on the concrete (visual sign of over-carbonation) is grounds for immediate termination of that run – operators are trained to observe this through the clear tent walls. All sensor data (CO<sub>2</sub>%, RH/T, IR temperature) are logged per second as part of the QA/QC record for each trial. These logs will be reviewed to ensure each test stayed within the intended window; any excursion (e.g. RH drop leading to drying) invalidates the test and it will be repeated after adjusting parameters.

**Dry-Ice Feeding & Personal Safety:** When handling dry ice for mix injection, small pellets (~10 mm size) are used for rapid sublimation. They are added to the mix only after all other ingredients are in and the mixer is about to be sealed – this minimizes open-air sublimation. The operator must wear insulated gloves, long sleeves, and a face shield (to guard against splinters of dry ice and intense cold). All observers stand clear during the initial seconds of mixing. If the mixer lid has a pressure relief, it will be checked to ensure it’s functioning (to vent excess gas and avoid overpressure). After the mixing period, the drum is opened cautiously and ventilated as described. Any solid CO<sub>2</sub> remaining (unlikely, as it will mostly have sublimated) is left to sublime in the hood – never sealed in a waste container. For the chamber curing, CO<sub>2</sub> cylinders are secured upright and equipped with a two-stage regulator and flow meter. Connections are leak-checked with a soap solution before each use. The flow rate is kept low (e.g. <2 L/min) during initial purge to prevent disturbing lightweight prints and to avoid over-pressurizing the tent. An oxygen depletion alarm is installed in the laboratory space: if ambient O<sub>2</sub> falls below 19.5% (indicating CO<sub>2</sub> buildup), audible and visual alarms will trigger, and the building HVAC will ramp up ventilation. Team members have been instructed in CO<sub>2</sub> safety: always work in pairs when entering an enclosed curing area and ventilate the space thoroughly before any close inspection of the specimens.

**Safe Setup and Shutdown:** Prior to each print or curing run, a safety checklist is followed. Key points: (1) Ventilation – lab exhaust fans on high, and doors open (if possible) when releasing CO<sub>2</sub>. (2) No ignition sources – while CO<sub>2</sub> is non-flammable, the procedure avoids any confusion with pressurized gas handling protocols (standard compressed-gas safety applies). (3) Emergency stop – the CO<sub>2</sub> supply has a clearly labeled shut-off valve accessible outside the tent, and the printer power has an emergency cut-off in case of any abnormal event. After CO<sub>2</sub> curing, the concrete specimens are moved to ambient curing or water curing as needed. The chamber tent is purged with fresh air (fans blowing in) for several minutes before opening fully. DO NOT enter an enclosed cure chamber immediately after high-CO<sub>2</sub> curing – the area is first tested with a handheld CO<sub>2</sub> meter to ensure levels are back to near normal (<0.5%). Finally, all gas cylinders are closed at the main valve after use, and pressure in regulators bled off. Any ice or condensate on equipment (from cold CO<sub>2</sub> expansion) is wiped dry to prevent slip hazards. By adhering to these SOPs, the team ensures that the innovative use of CO<sub>2</sub> in printing poses minimal risk to personnel and facilities while maintaining full control over the curing process.

## - Appendix E : LCA Assumptions & Calculation Log



**System Boundary & Declared Unit:** The life-cycle assessment will be cradle-to-gate, encompassing raw material production (cement, aggregates, admixtures), mix preparation and printing, and the CO<sub>2</sub> curing process up to the point the element is ready for use. The declared unit is 1 m<sup>3</sup> of 3D-printed concrete (structural element) cured under the new CO<sub>2</sub> process, as this provides a functional basis to compare against 1 m<sup>3</sup> of conventionally cured printed concrete. All inputs and outputs are tracked per cubic meter. The boundary includes cement manufacture (with its clinker factor and associated CO<sub>2</sub> emissions), production of SCMs (e.g. calcination energy for metakaolin if used), aggregate mining and transportation, mixing energy, and CO<sub>2</sub> capture and delivery. It stops at the factory gate – so downstream aspects like use-phase and end-of-life are excluded (though a carbonate stability assumption is noted for end-of-life). By focusing cradle-to-gate, we capture the embodied carbon and the immediate impact of CO<sub>2</sub> curing on that footprint.

**CO<sub>2</sub> Source, Capture, and Transport Assumptions:** We assume the CO<sub>2</sub> used for curing is sourced from an industrial waste stream (e.g. a fertilizer plant) and would otherwise be emitted to atmosphere – thus the CO<sub>2</sub> itself is treated as having zero “biogenic” or anthropogenic emission cost (i.e. we don’t count its chemistry as new carbon). However, the energy required to capture, liquefy, and transport that CO<sub>2</sub> is included. For a baseline, we use an estimate of 0.1–0.2 kWh of electricity per kg CO<sub>2</sub> for compression and cooling (assuming bulk CO<sub>2</sub> supply) and a trucking distance of 100 km in insulated cylinders. These inputs add a small carbon penalty per kg CO<sub>2</sub> used, which is accounted for. In one scenario, we will also examine direct air capture (DAC) CO<sub>2</sub> (which has a very high energy cost ~1 kWh/kg CO<sub>2</sub>) just for sensitivity, although it’s not the primary source. It’s assumed CO<sub>2</sub> curing uses approximately 5–15 kg CO<sub>2</sub> per m<sup>3</sup> concrete (depending on the dose band: low, mid, high). For each test, the exact CO<sub>2</sub> consumed (mineralized) is measured via mass gain/TGA, and any excess CO<sub>2</sub> vented is not counted as sequestered. Transportation of CO<sub>2</sub> is modeled using a diesel truck (ton-km) factor for the cylinder delivery. All these assumptions are logged in the LCA calculation spreadsheet (“CO2\_LCA\_Log.xlsx”) for transparency.

**Mix Design and Production Inputs:** The concrete mix is a Portland-limestone cement (PLC) based mortar with typical 3DCP admixtures. We model cement impacts using EPD data for CSA GU or Type IL cement (with ~10% limestone) adjusted for our clinker content. If SCMs are used (e.g. slag or fly ash), their “upstream” burdens are included (slag assumed to carry the grinding energy and a small allocation from blast furnace, fly ash is treated as industrial byproduct with minimal burden, aside from processing). Superplasticizer and other additives are included at their production impact per kg. Mixing energy is based on the electrical energy of our mixer and pump during a print (monitored via a power meter – roughly on the order of 5–10 kWh per m<sup>3</sup>, which is small but counted). The printing process (robot motion) electricity is also included (estimated from machine specs). These are summed in the cradle-to-gate inventory.

**Carbonation Curing Process in LCA:** The CO<sub>2</sub> curing itself has two opposite contributions in the carbon balance. On the emissions side, we include any energy for CO<sub>2</sub> curing (e.g. the IR heating or humidification if used, any fans, sensors – though minor, we include them for completeness). On the savings side, we credit the CO<sub>2</sub> sequestered permanently in the concrete. Following ISO 21930 and EN 15804 guidelines, we can subtract the amount of CO<sub>2</sub> mineralized in the product from the net GHG emissions of that product, provided it is expected to remain locked for the building’s life (100+ years). In our case, CO<sub>2</sub> is stored as thermodynamically stable calcium carbonates (calcite/aragonite) in the hardened matrix, which will not re-emit under normal use. Thus, we apply a negative CO<sub>2</sub> emission for the kilograms of CO<sub>2</sub> uptake measured (e.g. if 10 kg CO<sub>2</sub> are stored per m<sup>3</sup>, that’s –10 kg CO<sub>2</sub> in the emission tally for that scenario). However, we will also run a sensitivity where, if the element is demolished and crushed, some fraction of carbonates might re-carbonate or release CO<sub>2</sub> over end-of-life (e.g. in an acidic environment). For now, the base assumption is full permanent storage (100% credit) as a conservative stance in line with other carbonation cure studies. The LCA log will clearly state the credited CO<sub>2</sub> amount and any assumed timing (we assume the credit at the moment of curing, since that’s within the system boundary of cradle-to-gate for storage).

**Credits and Co-product Allocation:** No co-products are produced in our system except the primary concrete element. If any material savings occur (for instance, if CO<sub>2</sub> curing allows a lower cement content for the same strength – a hypothesis to test), we would account for that by reducing cement input for equivalent performance. That kind of improvement would appear as a reduced impact in the CO<sub>2</sub>-cured mix scenario. We do not generate excess energy or reusable byproducts to credit. The only “credit” comes from the carbon sequestration in the product itself (sometimes termed negative emissions or carbon storage). We will ensure to follow the ILCD/EN 15804 rules for declaring stored carbon: it will

be reported separately in the LCA results (as kg CO<sub>2</sub> sequestered). If the print has reinforcement (none in Phase 1–2 tests, but possibly steel fibers in Phase 3 element), the additional emissions for steel (and any change in its production due to CO<sub>2</sub> curing effects, likely none) are included as well.

**LCA Calculation Log:** All calculations are documented in a stepwise fashion in the provided Excel log. For each mix design and curing schedule, the log shows raw material quantities (kg), their embodied CO<sub>2</sub> from databases, process energy inputs (MJ or kWh converted to CO<sub>2</sub> using regional electricity factors), transportation distances, and finally the CO<sub>2</sub> uptake credit. Intermediate values like “effective CO<sub>2</sub> curing energy per m<sup>3</sup>” and “CO<sub>2</sub> cured concrete net CO<sub>2</sub>” are shown. The log also includes a segment for same-schedule comparison, meaning we align the age at testing for fair comparison (since our claim is carbon curing doesn’t sacrifice performance, we ensure we’re comparing equal-strength designs or same curing durations). The cradle-to-gate global warming potential (GWP) is the primary indicator, though we will also note if there are changes in other indicators (e.g. if CO<sub>2</sub> curing eliminates the need for steam curing, we also credit the avoided steam energy and its emissions). For instance, if a steam-cured reference uses 5 kWh of heat per m<sup>3</sup>, the CO<sub>2</sub>-cured version avoids that – we include that avoidance as well. Such assumptions are clearly annotated in the log file.

The LCA assumes a fair accounting where any extra emissions for implementing CO<sub>2</sub> curing (gas production, transport, power) are added, and any carbon stored, or energy saved are subtracted. The goal is to truthfully compute the net carbon footprint per unit of CO<sub>2</sub>-cured printed concrete, ensuring that we only claim a \*\*“carbon-negative” or “carbon-saving” outcome if the math (within these boundaries) supports it. The detailed assumptions and calculations will be available for review in Appendix tables, and key results will be used to support the feasibility of the process in the proposal.

## - **Appendix F: Calibration & Threshold Pack**

### ▪ **UPV to early-age stiffness and print timing**

**Purpose.** Use UPV only as a timing beacon so we deposit layers at the right stiffness. We do not claim UPV equals strength. Strength is still from tests.

**Specimens and schedule.** Make small cubes or cylinders from the same batch you will print. Take UPV at about 3 hours, 6 hours, 9 hours, and 24 hours. At 24 hours, test companion pieces in compression so the curve is anchored to a real point.

### **How we build our curve.**

1. Set up the UPV probes the same way every time. Use the same couplant. Wipe surfaces clean.
2. Record at least three readings per time point and keep the average.
3. Plot UPV against time on one page. Add your 24-hour strength point on the same sheet for reference.
4. Keep the smooth curve that fits the points well. If one point looks wrong, repeat it once and keep the median of the three tries.

### **How we turn this into print timing.**

- Run one or two small bench prints and note the time (or UPV) when the bead holds shape, does not slump after 15–30 minutes, and still bonds well to the next layer.
- Mark this as your **print timing band** (for example: “print when UPV is roughly between X and Y” or “print between 50 and 80 minutes after batching” if that is more practical).
- Write this band on the day’s build card. During runs, if we are **below** the band we wait; if we are **above** the band we shorten layer height or stop, because bond can suffer when we print too late.

**What we store.** The one-page UPV curve with the timing band, the raw readings, date, mix ID, and who took the readings. Put the PDF or image in the run folder.

### ▪ **ATR-IR and TGA “pass” thresholds for carbonation**

**Purpose.** Make a simple pass call from spectra and thermogravimetry so we do not promote a schedule that is under-carbonated or over-carbonated.

### **Sampling.**

Early screen at about 2 hours: take a thin surface sample (2–3 mm).

Decision point at about 24 hours: take both a surface sample and a mid-depth sample.

### **ATR-IR pass rules.**

At about 2 hours, the strong carbonate band near 1410 to 1470 should be clearly visible and not “faint”. If it is faint, we hold or re-run.

At about 24 hours, the carbonate band is strong at mid-depth and does not jump much compared with the 2-hour check. This means we are close to a plateau, not only a skin effect.

### **TGA pass rules.**

At about 24 hours, the portlandite loss band is small. That means most portlandite has reacted.

At about 24 hours, the carbonate loss band is present and not marginal. As a rough anchor, the carbonate mass loss should be at least a couple of percent of binder, or clearly above the control.

### **Promote, hold, drop.**

**Promote** when both ATR-IR and TGA look right and there is no chalky surface and no new microcracking.

**Hold** when ATR-IR looks good, but TGA is only just there. Re-check once. If still weak, reduce dose or contact time and try again.

**Drop** when ATR-IR stays weak at 24 hours, when portlandite is still clearly present, or when a chalky skin appears.

**What we store.** A single-page report with the two ATR-IR screenshots (surface and mid-depth) and the TGA curve with the bands highlighted, plus the sampling photos.

### **▪ Short RCPT surrogate and how we keep it honest**

**Purpose.** Use a quick transport indicator at early age, but only when we have proof it tracks the full test.

### **How we prove it.**

Pick six to eight mixes from your past work, including at least one slag-rich and two printable mortars.

For each, plot the short test value against the proper migration or RCPT value measured at the standard age.

Keep the simple best-fit line with a clear plot and the error written on the page in words (for example: “the short test predicts the standard within about ten percent inside this range”).

### **Where we use it.**

In Micro and Meso stages we report the short test as percent change versus the printable control on the same day.

In Macro we use the short test only when the result lies inside the proven range. If it is outside the range, we run the standard test.

**What we store.** One page with the scatter plot and the simple sentence about the fit quality. A short table that lists the mixes and their values.

### **▪ Sensors, alarms, and how we prevent drift**

**Set-up.** We use a CO<sub>2</sub> sensor in the tent, a second CO<sub>2</sub> or oxygen alarm in the lab for safety, a combined humidity and air temperature sensor in the tent, and an infrared camera on the specimen surface. We log at one reading per second and save the CSV file in the run folder. We take a photo of the infrared screen at the highest temperature during the run and save it in the folder.

**Emissivity for the infrared camera.** Place a small matte tape patch near the region of interest and set the camera so the concrete and the tape read the same number. Keep this setting for the day.

### **Alarms and caps.**

CO<sub>2</sub> inside the tent holds near ten percent. Alarms if it falls well below or rises well above that band.

Humidity stays near the mid-sixties. Alarms if it drops below the high fifties or goes above the mid-seventies.

Surface temperature should not climb more than a few degrees above room temperature and not exceed about thirty degrees overall. If it does, we stop gas, vent the tent, and log the cause.

If any sensor drifts or freezes, we void the run, fix the sensor, and repeat. The voided log stays in the folder with “sensor drift” noted.

**What we store.** One page with the graph of CO<sub>2</sub>, humidity, and temperature over time, the infrared photo at peak, and the checklist with all boxes ticked or crossed.

#### ▪ Ready-to-use forms (what each page will contain)

**Print and cure log (per run).** Specimen ID, date and time, mix ID, dry-ice dose, contact time, tent CO<sub>2</sub> (if used), humidity and air temperature, infrared peak temperature, any abort, short note on what changed.

**Geometry and buildability.** Target bead width and height, measured values at placement and at 15–30 minutes, wall target height, achieved height, failure mode if any (buckle, slide, collapse), a box for “pass” or “repeat”.

**Interlayer bond.** Specimen ID, test type (pull-off or slant-shear), test area or coupon size, peak load or stress, failure mode (glue, substrate, or interface), ratio versus printable control, a box for “pass” or “repeat”. Glue or substrate failures do not count as bond results.

**Durability mini-panel.** Absorption at 24 hours, transport value from the short test, carbonation depth check if used, all reported as less than or equal to the printable control, a box for “pass” or “repeat”.

**Pass sheet.** Geometry pass, bond pass, durability pass, no chalking, infrared cap respected, sensors within bands, sign-off by tester and witness.

#### ▪ Dry-ice safety and best practice (primary route)

**Before you start.** Wear cryogenic gloves, eye and face protection, long sleeves. Weigh pellets quickly and keep them in an open tray so gas can vent. Seal the mixer lid before the pellets fully sublime. Stand clear for the first seconds of mixing.

**After mixing.** Open the mixer in a fume hood or a space with strong air flow. Let gas clear before anyone looks inside. Do not trap dry ice in a closed bin. Let it fade in the hood.

**For prints with a tent.** Use low and steady gas flow so filaments are not disturbed. Keep humidity stable. Never climb inside a tent full of CO<sub>2</sub>. Always vent and verify safe air before any close work.

#### ▪ Quality check points in plain words

1. We only move a schedule forward when the bead and wall stay within the target size band and the wall reaches target height without a failure.
2. We only move a schedule forward when interlayer bond is the same as the printable control or higher.
3. We only move a schedule forward when the absorption and the transport indicator are the same as the printable control or better.
4. We stop and adjust if we see chalking, if the surface overheats, or if sensors go out of band.
5. We only claim carbon credit when the element passes all of the above and we have measured storage from ATR-IR and TGA for that schedule.