

**LabCim – Laboratório de Cimentos | UFRN**  
Carbon Depth®

## **Phenolphthalein-Based Method for the Determination of Carbonation Depth in Cementitious Materials**

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### **Document Information**

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### **⚠ Safety warning**

Before initiating this procedure, strict compliance with all applicable laboratory safety requirements is mandatory. Appropriate Personal Protective Equipment (PPE) must be worn at all times, including laboratory coat, safety goggles, protective gloves, and any additional protection required by the specific task.

All operational limitations related to temperature, pressure, and volume capacity of materials and equipment must be carefully observed. This procedure may involve the handling of potentially hazardous chemicals and, in certain cases, the operation of systems under elevated temperature and/or pressure conditions, which may pose risks of chemical exposure, burns, mechanical failure, or physical injury.

This test shall be performed only by trained and authorized personnel who are familiar with the associated hazards and emergency procedures.

Failure to comply with these safety requirements may result in serious injury or equipment damage.



## 1. General Information

The phenolphthalein alkalinity test is widely employed to estimate the carbonation depth in hardened cementitious materials. The method is based on the pH-sensitive colorimetric response of phenolphthalein, which exhibits a violet/pink coloration in highly alkaline environments (typically pH > 9.5–10.0) and remains colorless at lower pH values (approximately pH < 8.2), characteristic of carbonated regions.

Carbonation occurs as atmospheric carbon dioxide reacts with calcium hydroxide and other alkaline phases within the cement matrix, resulting in a reduction of pore solution alkalinity. When phenolphthalein solution is applied to a freshly fractured surface, the contrast between the colored (non-carbonated, alkaline) zone and the uncolored (carbonated) zone becomes clearly visible. The interface between these regions defines the carbonation front.

Traditionally, carbonation depth is determined by measuring the distance between the exposed surface and the color transition boundary using a caliper.

However, the identification of the carbonation front is inherently operator-dependent, particularly in specimens exhibiting heterogeneous pore structure, irregular fracture surfaces, or gradual color transitions. In practice, significant inter-analyst variability may arise due to subjective interpretation of the color boundary, especially when the measured depths range between 0.5 and 8 mm. Differences of several millimeters between operators are not uncommon, potentially compromising repeatability and comparability of results.

The implementation of standardized image acquisition and automated digital processing reduces subjective bias, enhances reproducibility, and provides objective quantification of the carbonation front.

The **Carbon Depth®** program was developed to convert this conventionally semi-quantitative visual method into a quantitative analytical platform. Through standardized image acquisition and digital image processing, the software enables automated determination of carbonation depth, calculation of the carbonated area fraction, and estimation of the carbonated volume of the specimen.

In addition, the platform supports carbonation kinetics analysis by correlating depth measurements with exposure time, allowing the fitting of diffusion-based models (e.g.,  $X = k\sqrt{t}$ ) and facilitating the quantitative assessment of carbonation rates under natural or accelerated conditions.

## 2. Accelerated Carbonation Test Conditions

At LabCim, carbonation experiments are typically conducted under accelerated conditions using pressurized reactors designed to simulate environments representative of oil and gas wells. The objective is to evaluate the carbonation depth in cement paste, mortar, or concrete specimens exposed to CO<sub>2</sub>-rich atmospheres under controlled thermo-hygrometric and pressure conditions.

The procedure follows the fundamental principles established in RILEM CPC-18 and EN 14630:2006, with necessary adaptations for operation under elevated pressure. Particular attention shall be given to the following parameters:

- Carbon dioxide environment: Exposure to a CO<sub>2</sub>-rich atmosphere representative of the target application (e.g., wellbore or reservoir conditions);
- Temperature control: Specimens shall be maintained at predefined temperatures consistent with the experimental design;
- Pressure conditions: Operating pressure shall be selected to simulate downhole conditions or other relevant service environments, according to the capabilities of the reactor system;
- Relative humidity: Humidity shall be controlled to prevent excessive surface drying and to ensure conditions conducive to carbonation reactions;
- Exposure duration: Predefined exposure periods may range from short-term intervals (e.g., 7, 14, 28, and 90 days) to extended durations corresponding to accelerated long-term simulations (e.g., 180 days and multi-year equivalent exposures), depending on the adopted acceleration and extrapolation model.

All experimental parameters shall be recorded and reported to ensure reproducibility and traceability of results.

## 3. Cutting and Specimen Preparation

Proper specimen preparation is critical to ensure accurate determination of carbonation depth. All procedures shall be conducted in a manner that preserves the integrity of the carbonation profile and prevents artificial alteration of the exposed surface.

### 3.1 Timing of Sectioning

Specimens shall be sectioned only after completion of the predefined carbonation exposure period, in accordance with the experimental plan.

### 3.2 Cutting Procedure

Sectioning shall be performed using a diamond blade. Excessive frictional heating during cutting may locally modify the chemical profile of the cementitious matrix, potentially affecting the carbonation front.

Whenever feasible, freshly fractured surfaces are preferred over sawn surfaces for subsequent indicator application, as they better preserve the original carbonation boundary and minimize thermal or mechanical disturbance.

### **3.3 Minimum Section Thickness**

A minimum cross-sectional thickness of 25 mm is recommended, consistent with RILEM CPC-18 guidelines, to reduce edge effects and unintended CO<sub>2</sub> ingress from lateral surfaces.

### **3.4 Surface Conditioning**

Immediately prior to indicator application, the exposed surface shall be gently dried using absorbent paper to remove surface moisture. The use of hot air, ovens, or direct heat sources is strictly prohibited, as temperature elevation may influence indicator response and local moisture distribution.

### **3.5 Surface Cleaning**

Loose particles, dust, and debris shall be removed using dry compressed air or a clean, dry brush. Washing with water or any abrasive treatment shall be avoided to prevent alteration of the carbonation boundary.

## **4. Phenolphthalein Indicator Solution**

The phenolphthalein indicator solution is used to visually identify the carbonation front by differentiating between alkaline (non-carbonated) and carbonated regions within cementitious materials.

### **4.1 Preparation of the Indicator Solution**

The solution shall be prepared as follows:

- Dissolve 1.0 g of phenolphthalein in 70 mL of absolute ethanol ( $\geq 99\%$ );
- Dilute to a final volume of 100 mL using deionized or distilled water;
- Mix thoroughly to ensure complete dissolution and homogeneity;
- Store the solution in an amber glass bottle, tightly sealed, to prevent photodegradation and solvent evaporation.

The solution should be gently agitated prior to each use to maintain uniformity.

Alternative alcohol-based solvents (e.g., 95% ethanol or isopropanol) may be used when absolute ethanol is unavailable, provided that the indicator response is not affected.

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## 4.2 Indicator Transition Range

Phenolphthalein exhibits a color transition within a narrow alkaline range:

- Colorless at  $\text{pH} \leq 8.2$
- Pink to purple at  $\text{pH} \geq 8.3$

In cementitious systems, non-carbonated regions (high alkalinity, typically  $\text{pH} > 10$ ) develop a pink–purple coloration upon application, whereas carbonated regions remain colorless due to the reduction in pore solution pH.

## 5. Indicator Application and Measurement

### 5.1 Application of the Indicator Solution

The phenolphthalein solution shall be applied to a freshly fractured or properly cut, clean, and dry surface.

- The indicator shall be applied using a fine mist spray bottle or a soft-bristle brush to ensure uniform surface coverage.
- Excess solution shall be avoided, as runoff may obscure the carbonation boundary and compromise visual interpretation.
- After application, allow approximately 30 seconds for color stabilization prior to measurement, consistent with established testing guidelines.

The color transition boundary between the pink–purple alkaline zone and the colorless carbonated zone shall be clearly identified before proceeding with measurements.

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### 5.2 Measurement of Carbonation Depth

- Carbonation depth shall be measured at 6 to 12 locations distributed along the exposed surface to ensure statistical representativeness.
- At each location, the depth of the colorless (carbonated) zone shall be measured using a calibrated digital caliper.
- Measurements shall be taken perpendicular to the exposed surface.

- Depth values below 0.5 mm shall be disregarded, as recommended in RILEM CPC-18, due to limitations in measurement precision and surface variability.
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### 5.3 Data Recording and Final Results

- The arithmetic mean of all valid carbonation depth measurements shall be reported as the representative carbonation depth.
- When relevant, the maximum local depth ( $d_{k\max}$ ) may also be reported to document isolated regions of increased penetration.
- The analyzed surface shall be documented photographically using a camera with a minimum resolution of 12 megapixels.
- Image files shall be systematically named to include specimen identification, carbonation exposure time, and replicate number.

For improved reproducibility of image analysis, the use of a portable photographic lightbox with integrated LED lighting is recommended. Controlled lighting conditions (neutral background, uniform lateral illumination, and adequate diffusion) are essential to ensure reliable visual interpretation and subsequent digital image processing.



## 6. Digital Image Analysis Using Carbon Depth®

### 6.1 Rationale for Digital Quantification

Traditionally, carbonation depth is determined by manual measurement of the color transition boundary using calipers. Although widely adopted, this approach is inherently operator-dependent and susceptible to inter-analyst variability, particularly when the carbonation front presents irregular geometry or diffuse color transitions.

In practice, differences of several millimeters may occur between analysts when total depths range between 0.5 and 8 mm, which may significantly affect interpretation of durability performance and service-life prediction.

Carbon Depth® was developed to provide:

- Objective and reproducible measurement of carbonation depth
  - Automated quantification of carbonated area fraction
  - Estimation of carbonated volume
  - Statistical treatment of measurements
  - Kinetic modeling of carbonation progression
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## 6.2 Image Acquisition and Upload

- Images shall be uploaded in .png, .jpg, or .jpeg format.
- Photographs shall be acquired under controlled lighting conditions as described in Section 5.3.
- A neutral (preferably white) background is recommended to improve segmentation stability.

The current version incorporates enhanced segmentation stability and quality assessment routines while preserving the validated core processing algorithm.

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## 6.3 Specimen Configuration

Users must define the specimen geometry:

- Cylindrical: diameter and height (mm)
- Rectangular/prismatic: width, depth, and height (mm)

These dimensions are required for volumetric calculations and scaling of pixel-to-millimeter conversion.

Future versions may include optional two-point calibration for image-based dimensional scaling without modifying the validated core segmentation logic.

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## 6.4 Segmentation and Morphological Refinement

Carbon Depth® performs automated segmentation of the alkaline (purple) region using color-space discrimination and morphological refinement.

The user may adjust:

- Cleanup kernel (alkaline region smoothing)

This refinement step reduces noise and improves boundary definition without altering the fundamental segmentation model.

The current version includes enhanced robustness to:

- Uneven illumination
  - Minor surface artifacts
  - Color saturation variability
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## 6.5 Quantitative Outputs

The software automatically provides:

- Original image display
  - Segmented alkaline mask
  - Carbonated area fraction (%)
  - Estimated carbonated and non-carbonated volumes
  - Multi-point depth estimation
  - Mean carbonation depth
  - Standard deviation
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## 6.6 Kinetic Analysis and Modeling

Carbon Depth® includes tools for carbonation kinetics analysis when measurements at multiple exposure times are available.

Carbonation progression in cementitious materials is commonly described by diffusion-controlled models, frequently approximated by the square-root-of-time relationship:

$$x = k\sqrt{t}$$

where:

- $x$ = carbonation depth
- $t$ = exposure time

- $k$ = carbonation coefficient

This formulation derives from Fick's second law under simplified boundary conditions and is widely adopted in durability studies (e.g., RILEM CPC-18; EN 14630; fib Model Code).

The software enables regression analysis to estimate the carbonation coefficient and evaluate goodness-of-fit (e.g.,  $R^2$ ), supporting service-life assessment and comparative durability studies.

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## 6.7 Quality Assurance (QA Flags)

To enhance reliability, the system performs automatic quality checks, including:

- Detection of abnormally small alkaline mask area
- Identification of fragmented contours
- Low illumination warning
- Segmentation instability indicators

These alerts do not override results but inform the user of potential reliability concerns.

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## 6.8 Report Generation

The program allows generation of a structured PDF technical report including:

- Method description
- Specimen information
- Depth measurements and statistics
- Area and volume quantification
- Kinetic modeling results (when applicable)
- Embedded original and processed images

All outputs are designed to ensure traceability and reproducibility.

## 7. Important Considerations and Sources of Uncertainty

To ensure reliability and reproducibility of results obtained using the phenolphthalein carbonation test, the following technical considerations shall be observed.

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### 7.1 Timing Between Sectioning and Indicator Application

Excessive delay between specimen cutting and indicator application shall be avoided. Prolonged exposure of freshly cut surfaces to ambient air may result in superficial, spurious carbonation due to atmospheric CO<sub>2</sub> uptake. Such secondary reactions may artificially alter surface pH and compromise accurate identification of the true carbonation front.

#### Temporary Storage

If immediate application of the indicator is not possible, specimens shall be stored:

- In a sealed, airtight container; or
- In a CO<sub>2</sub>-free atmosphere

to prevent surface pH alteration prior to testing.

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### 7.2 Environmental Conditions During Testing

Testing under extreme environmental conditions may affect indicator response and color stability.

- Relative humidity above 90% may dilute surface alkalinity and impair color development.
- Relative humidity below 40% may promote rapid drying and non-uniform indicator distribution.

For laboratory-controlled testing, a relative humidity between 60% and 70% is recommended to ensure consistent chromatic response.

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### 7.3 Special Considerations for Modified Concretes

Cementitious materials treated with hydrophobic agents, silanes, siloxanes, or polymer modifiers may exhibit delayed or attenuated phenolphthalein response due to altered pore solution accessibility.

In such cases, complementary techniques (e.g., petrographic analysis or thermogravimetric analysis) may be required to confirm carbonation depth.

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#### **7.4 Common Sources of Error**

The most frequent sources of experimental error include:

- Excessive heating during cutting: Dry cutting or inadequate cooling may locally modify the chemical profile of the matrix, potentially inducing false coloration.
  - Wet or washed surfaces: Surface moisture may dilute the indicator or suppress chromatic development, impairing boundary detection.
  - Excessive application of indicator solution: Runoff may create artificial color zones not representative of the actual carbonation front.
  - Inadequate illumination during imaging: Uneven lighting, shadows, glare, or non-neutral backgrounds may compromise automated segmentation and quantitative analysis in Carbon Depth®.
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#### **7.5 Criteria for Repetition of the Test**

The test shall be repeated under the following conditions:

- Diffuse or poorly defined coloration boundaries preventing objective measurement;
- Absence of coloration in regions known to remain alkaline;
- Physically inconsistent results (e.g., negative depth values or discrepancies exceeding 50% between opposing sides of the specimen);
- Evidence of contamination or improper handling prior to indicator application;
- Inadequate image acquisition quality (e.g., insufficient resolution, glare, improper focus) when digital processing is intended.

## References

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