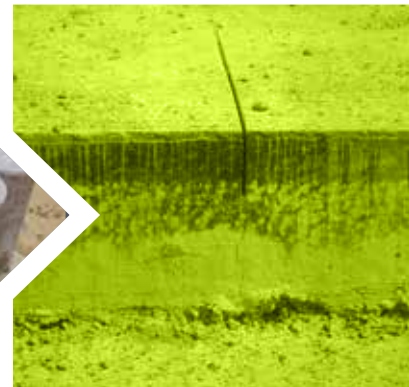
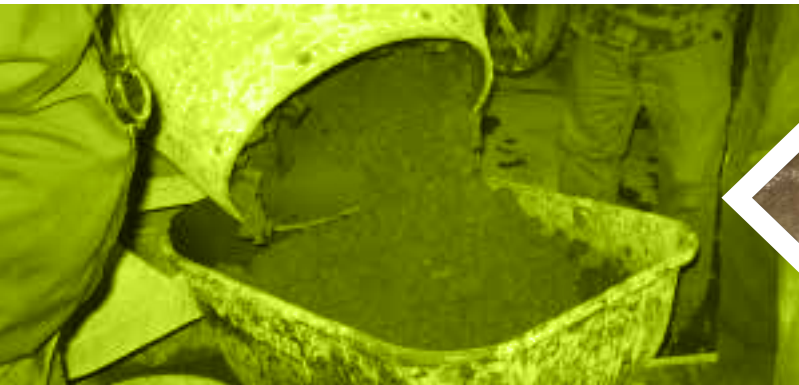




EXPLORATORY ADVANCED RESEARCH

Mechanisms of Hydration and Setting of Ordinary Portland Cement in Simple and Complex Systems

RESEARCH SUMMARY REPORT



U.S. Department of Transportation
Federal Highway Administration

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Technical Report

Documentation Page

1. Report No. FHWA-HRT-17-102		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Mechanisms of Hydration and Setting of Ordinary Portland Cement in Simple and Complex Systems				5. Report Date April 2019	
7. Author(s) Rolf Arvidson, Jeffrey Bullard, Josephine Cheung, Brad Chmelka, Tyler Ley, Larry Roberts, George Scherer, Denise Silva, Patrick Zickler				6. Performing Organization Code	
9. Performing Organization Name and Address Princeton University 4 New South Princeton, NJ 08544-0036; National Institute of Standards and Technology 100 Bureau Drive Gaithersburg, MD 20899 TMNcorp 131 Rollins Ave., Unit 4A Rockville, MD 20852				8. Performing Organization Report No.	
				10. Work Unit No.	
				11. Contract or Grant No. DTFH61-12-H-00003, DTFH61-13-X-30003, DTFH61-15-A-00003	
12. Sponsoring Agency Name and Address Office of Corporate Research, Technology, and Innovation Management Federal Highway Administration 6300 Georgetown Pike McLean, VA 22101-2296				13. Type of Report and Period Covered Research Summary Report July 2012 – June 2017	
				14. Sponsoring Agency Code	
15. Supplementary Notes: Contracting Officer's Representative: Richard Meininger, HRDI-10 Technical Contact: David Kuehn, HRTM-30					
16. Abstract This research summary report provides a description of research carried out to improve the understanding of the mechanisms of hydration of portland cement in complex and simple mixtures; develop analytical methods to directly observe hydration processes in real time; and develop and validate improved computer models to design optimal concrete composition, curing methods, performance, and durability. The project resulted in innovative analytical techniques that made it possible to perform direct micro- and nanoscale observation of reactions. This new perspective allowed researchers to develop a new and clearer hypothesis to understand the mechanisms of cement hydration. Computer models based on the new hypothesis will provide engineers and practitioners with tools to produce more efficient, durable, and cost-effective concrete products and structures.					
17. Key Words Concrete, portland cement, hydration, ordinary portland cement, cement hydration kinetics and modeling, advanced research, hydration mechanisms, setting of ordinary portland cement, hydration process, concrete performance, hydration breakthroughs, concrete composition.				18. Distribution Statement No restrictions. This document is available through the National Technical Information Service, Springfield, VA 22161. http://www.ntis.gov	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 20	22. Price N/A		

SI* (Modern Metric) Conversion Factors

APPROXIMATE CONVERSIONS TO SI UNITS

SYMBOL	WHEN YOU KNOW	MULTIPLY BY	TO FIND	SYMBOL
LENGTH				
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
AREA				
in ²	square inches	654.2	square millimeters	mm ²
ft ²	square feet	0.093	square meters	m ²
yd ²	square yard	0.836	square meters	m ²
ac	acres	0.405	hectares	ha
mi ²	square miles	2.59	square kilometers	km ²
VOLUME				
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	meters	L
ft ³	cubic feet	0.028	meters	m ³
yd ³	cubic yards	0.765	kilometers	m ³
NOTE: volumes greater than 1000 L shall be shown in m ³				
MASS				
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
TEMPERATURE (exact degrees)				
°F	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	°C
ILLUMINATION				
fc	foot-candies	10.76	lux	lx
ft	foot-Lamberts	3.426	candela/m ²	cd/m ²
FORCE and PRESSURE or STRESS				
lbf	poundforce	4.45	newtons	N
lbf/in ²	poundforce per square inch	6.89	kilopascals	kPa

APPROXIMATE CONVERSIONS FROM SI UNITS

SYMBOL	WHEN YOU KNOW	MULTIPLY BY	TO FIND	SYMBOL
LENGTH				
mm	millimeters	0.039	inches	in
m	meters	3.28	feet	ft
m	meters	1.09	yards	yd
km	kilometers	0.621	miles	mi
AREA				
mm ²	square millimeters	0.0016	square inches	in ²
m ²	square meters	10.764	square feet	ft ²
m ²	square meters	1.195	square yard	yd ²
ha	hectares	2.47	acres	mi
km ²	square kilometers	0.386	square miles	mi ²
VOLUME				
mL	milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	gallons	gal
m ³	cubic meters	35.314	cubic feet	ft ³
m ³	cubic meters	1.307	cubic yards	yd ³
MASS				
g	grams	0.035	ounces	oz
kg	kilograms	2.202	pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
TEMPERATURE (exact degrees)				
°C	Celsius	1.8C+32	Fahrenheit	°F
ILLUMINATION				
lx	lux	0.0929	foot-candies	fc
cd/m ²	candela/m ²	0.2919	foot-Lamberts	ft
FORCE and PRESSURE or STRESS				
N	newtons	0.225	poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in ²

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380. (Revised March 2003)

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Introduction

Concrete is the world's most commonly used man-made construction material and second only to water as the most consumed substance on Earth.¹ Concrete can be produced in a wide variety of shapes and textures and is extremely durable, with a service life of 50 years or more in many common applications. Importantly, concrete can be made on demand and on site using portland cement, water, and aggregate materials, such as sand, gravel, crushed stone, and recycled crushed blast furnace slag. When mixed with water, portland cement forms a slurry that stays in a fluid state for several hours. During this induction phase of hydration, concrete can be transported and poured, even though the mixture is undergoing chemical and structural changes. Following the induction phase, the concrete mixture stiffens and generates heat during an acceleration phase marked by nucleation and growth of calcium hydroxide (CH) and calcium silicate hydrates (C-S-H; the dashes indicate that the compounds do not have a fixed ratio of silicon to calcium). Following this phase, heat generation drops off, but concrete continues to cure and harden. Mixtures containing high percentages of portland cement reach maximum strength within a few months. More complex mixtures with pozzolans or slag cement may continue to strengthen slowly for several years.

Although the basic concrete recipe—a combination of water, cement, and aggregate—has been in use since the early 19th century, scientists and engineers do not fully understand the chemical reactions that drive hydration and are responsible for the hardening, strength, and durability of concrete. Because of the uncertainties associated with hydration, it has not been possible to develop computer models that accurately predict the properties of the final product or explain the specific contribution of the binding agents, alternative aggregate components, or other additives. As a result, concrete is typically overdesigned by at least 10 percent to ensure that the final product meets its design performance. A better understanding of hydration would allow engineers to lower the strength overdesign “cushion” by reducing

the portland cement content and thereby decreasing material costs by as much as 10 percent, without sacrificing performance, and potentially reducing volume change and cracking related to high cement content. Better computer models of hydration would also lessen concrete's environmental impact. Reduction in portland cement content will decrease the carbon dioxide (CO₂) footprint of concrete, since portland cement production releases a high amount of CO₂. Compared with other construction materials, concrete has a very low embodied CO₂ content—a measure of greenhouse gas associated with production. However, because so much concrete is used each year, it is responsible for about 8 percent of industrial CO₂ production in the United States.¹

The goal of the Federal Highway Administration's (FHWA) project, “Mechanisms of Hydration and Setting of Ordinary Portland Cement in Simple and Complex Systems,” was to develop more efficient and effective ways to use concrete. Project scientists developed innovative analytical technologies to observe the mechanisms of hydration in three dimensions at the nano-, micro-, and macroscopic scales. These unprecedented observations have provided a depth of understanding of hydration mechanisms that was never before possible. Researchers used this improved understanding of hydration mechanisms to develop and validate computer models that will improve concrete composition and performance. The project was funded through FHWA's Exploratory Advanced Research (EAR) Program. Princeton University conducted the research in collaboration with research partners at the National Institute of Standards and Technology (NIST); Oklahoma State University (OSU); Rice University; University of California, Santa Barbara (UCSB); and GCP Applied Technologies, formerly W.R. Grace and Company.



In 2009, the concrete research community held an international conference—the Summit on Cement Hydration Kinetics and Modeling—in Quebec, Canada, to discuss research needs related to cement hydration. U.S. sponsors of the summit included FHWA, NIST, and the National Science Foundation. Following the summit, NIST and FHWA issued a joint publication outlining their vision for needed research, *NIST Special Publication 1138: Paving the Way for a More Sustainable Concrete Infrastructure: A Vision for Developing a Comprehensive Description of Cement Hydration Kinetics*.² The EAR Program project, “Mechanisms of Hydration and Setting of Ordinary Portland Cement in Simple and Complex Systems,” advances the joint research vision identified at the summit and expressed in the joint publication: “... focused experimental and computational modeling research that will provide the knowledge and translate it to industry tools for improved cements, mixture design, and service life prediction of concrete in service.”

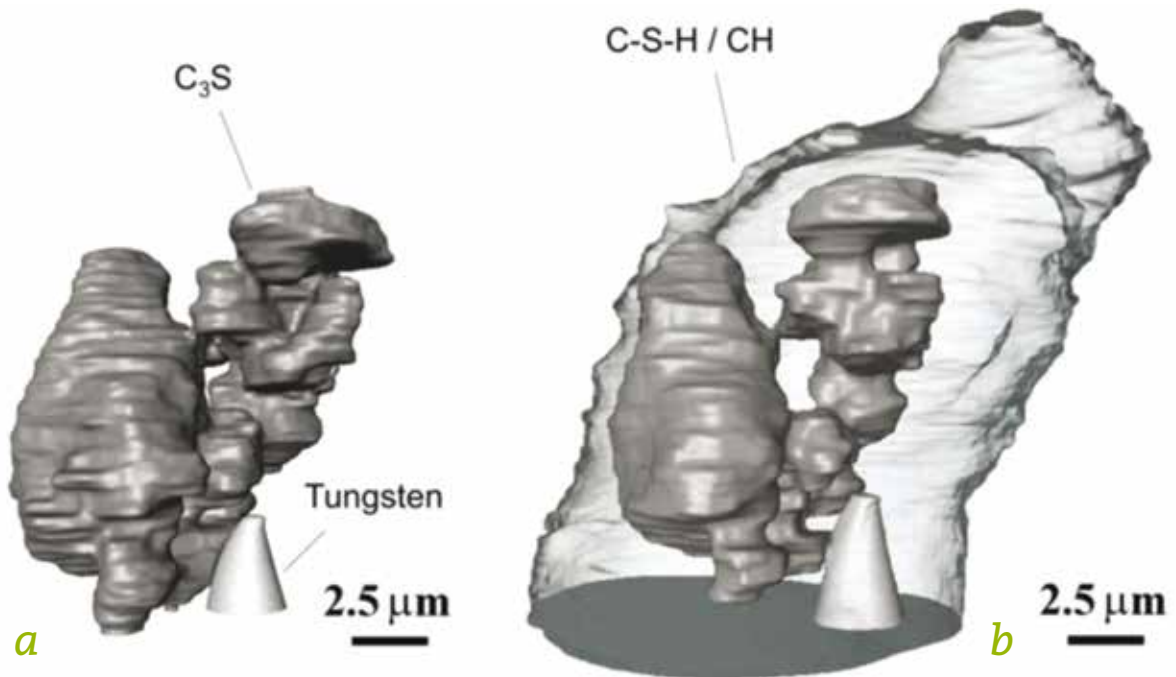


Figure 1. Illustration: Experimentally acquired tomographs. ((a) Initial particle configuration on a tungsten needle. (b) After 7 hours of reaction in an aqueous solution with initial CH concentration of 15 mmol L^{-1} . C_3S particles are dark gray, the tungsten needle is light gray, and the hydration products are shown as transparent.) ©The American Ceramic Society.³

Case Study: Using Advanced Analytical Techniques to Study Cement Hydration

Researchers at OSU focused on the use of advanced tomography and fluorescence imaging to experimentally visualize cement hydration at the micro- and nanoscales. The OSU team used a combination of nano-computed tomography (nCT) to capture the three-dimensional (3D) structure and nano x-ray fluorescence to reveal the 3D chemistry of cement before, during, and after hydration. This technique allowed the researchers to see changes in density and chemistry caused by hydration. Similar to x-ray computed tomography commonly used in medical sciences for nondestructive imaging of internal biological structures, nCT combines a series of images acquired at small angles of rotation around the target to produce a 3D model or tomograph. The relatively low-energy-level x-rays used

in nCT make it possible to construct 3D tomographs of cementitious and geologic materials. Interpretation of the images, and alignment of the images with the compositional data, required researchers to create the sophisticated software needed for analysis of huge quantities of data.

Using nCT, the OSU researchers directly observed the changing microstructure of tricalcium silicate (C_3S), a portland cement component that is primarily responsible for rapid hardening and plays an important role in the initial set and early strength of concrete. The research team used 3D microstructure imaging to make point-by-point comparisons of observed particle reactions and computer simulations of particle reactions in an aqueous

Case Study: Using Advanced Analytical Techniques to Study Cement Hydration (Cont.)

solution of CH at a resolution of 50 nm and sometimes as low as 15 nm depending on the experiment. The outputs of these analytical techniques developed at OSU have been used as a direct input in hydration models.

At UCSB, researchers focused on the development, demonstration, and use of new methods of nuclear magnetic resonance (NMR) spectroscopy to characterize at a molecular level the compositions, structures, and properties of cementitious materials. This characterization relied on combinations of powerful solid-state NMR capabilities, such as dynamic-nuclear-polarization (DNP) enhancement techniques, to identify and quantify surface species in hydrating cementitious materials with unprecedented sensitivity and resolution. The work has led to new, significant, and molecular-level insights into the detailed compositions, structures, and site-specific interactions of cementitious materials during hydration. Solid-state silicon-29 (^{29}Si), hydrogen-1 (^1H), calcium-43 (^{43}Ca), and magnesium-25 (^{25}Mg) NMR measurements were conducted on triclinic C_3S ,

monoclinic C_3S , tricalcium aluminate (C_3A), and white portland cements to identify and quantify the molecular species present, their transient formation or depletion under hydrating conditions, and estimated mean thicknesses of early hydration layers.

The DNP-NMR analyses examined the adsorption of retarder species like saccharides or phosphonates. The results enabled the molecular origins of retarder properties and the performances to be determined. This determination included the specific atomic-level interactions that account for competitive adsorption of dilute retarder quantities versus water at silicate surfaces, which dramatically slow the hydration of low-surface-area silicate particles. The UCSB researchers made key advances, including the use of 2D and DNP-enhanced NMR measurements, to yield a new understanding of the compositions and structures of hydrating cementitious materials for industrially relevant materials and formulations.

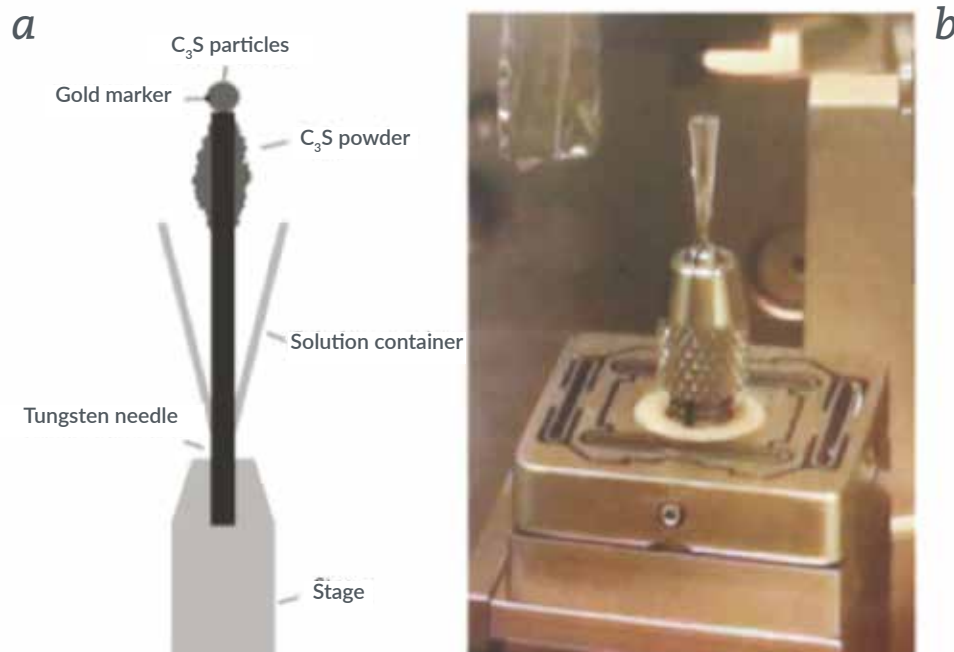


Figure 2. Illustration/Photo: The nano-computed x-ray tomography experimental setup. ((a) This is a diagram of the setup. (b) This setup has a cement C_3S particle on a tungsten needle in a solution that allows the particle to hydrate during the experiment.) ©The American Ceramic Society.³



Figure 3. Photo: A researcher determines the mass of a specimen on a laboratory balance. Source: FHWA.

Case Study: A Clearer, More Comprehensive Understanding of Hydration Mechanisms

The important analytical and imaging advances described above revealed features of the hydration process that had never before been directly observed. The rapid reaction with water creates pits in the surface of C_3S particles that quickly fill with hydration products having a high Ca/silicate ratio. One remarkable observation was that voids form in the paste after about 1 hour of hydration. At the start of the acceleration period, when the solute concentration drops because of rapid precipitation of C-S-H, the quantity of voids decreases and their size distribution changes. These voids influence the strength of the hardened paste.

The researchers have developed a new hypothesis to explain the induction and acceleration periods. This hypothesis is supported by numerous observations made at the nano- and microscale levels for this project:

1. Nanoscale measurements show that there are certain regions within C_3S particles, probably associated with crystalline defects, that dissolve more rapidly than others.

Case Study: A Clearer, More Comprehensive Understanding of Hydration Mechanisms (Cont.)

2. These regions form etch pits that are quickly filled with C-S-H, perhaps because of the high ion concentration in the pit.
3. As these fast-reacting regions continue to dissolve, the ions must diffuse through the newly formed C-S-H, so the ion concentration within the pores of the C-S-H will increase at a much higher rate than in the rest of the liquid in the cement mixture.
4. Soon, other solid products, such as portlandite, CH, begin forming within the pits.
5. The solids forming within the pores of the C-S-H reduce ion transport, slowing the reaction and causing the induction period. Ion concentrations continue to build in the bulk solution until they start forming solid hydration products such as CH.
6. Once these solid products begin to form, the concentration of ions in the solution drops and the nano-CH and sulfate from gypsum or hemihydrate ($\text{CaSO}_{4,2}\text{H}_2\text{O}$ or $\text{CaSO}_{4,1.2}\text{H}_2\text{O}$) dissolve.
7. This opens the pores of the C-S-H and allows rapid reaction of the fast-reacting regions, resulting in the start of the acceleration period.

Figure 4. Photo: Workers place concrete with a slip form concrete paver. Source: FHWA.



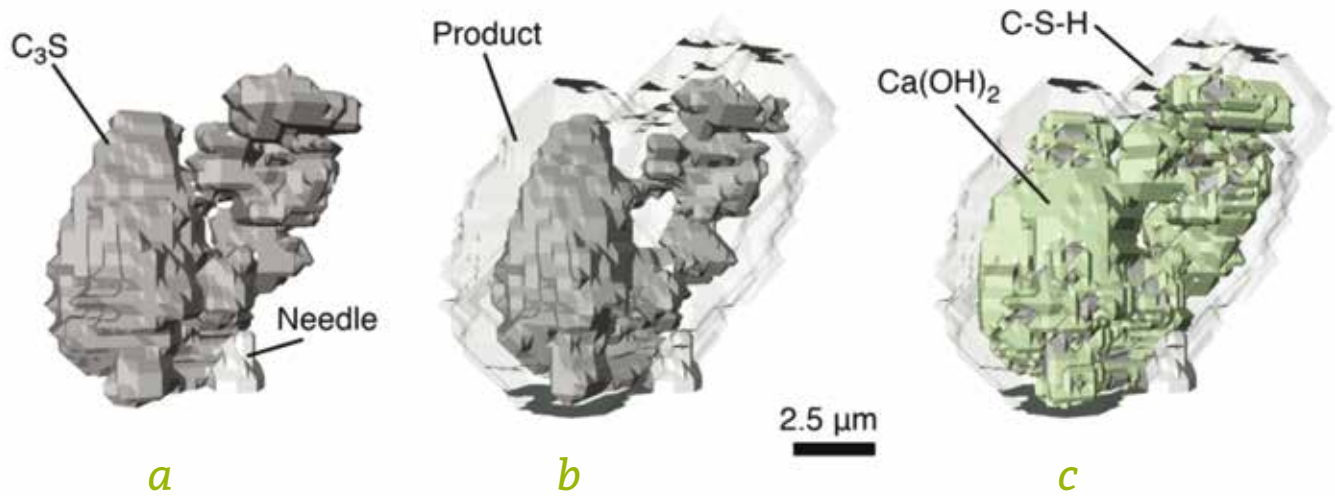


Figure 5. Illustration: Results of a HydratiCA run using the best available material and reaction data. ((a) The initial sample before hydration, a coarse-grained replication, voxel-by-voxel, of the system observed by the OSU group. (b) Changes in the C_3S particles after 7 hours of simulated hydration, with the hydration product indicated as a transparent shell form in comparison to figure 1. (c) The simulated distribution of $Ca(OH)_2$ [green] and C-S-H [transparent].) ©The American Ceramic Society.³

Case Study: New Models to Predict Concrete Performance

Researchers with the NIST model development group used observational findings to advance the state of cement hydration models and develop a validated, user-friendly engineering tool that could reliably predict the performance of concrete in the field and provide insights into how to manipulate that performance. The primary focus of the NIST research activities was to improve the simulation performance of HydratiCA, a kinetic cellular automaton model based on fundamental chemical reaction kinetic principles. HydratiCA is verified for some simple cementitious reactions but has historically lacked experimental measurement data needed for simulations of more complex hydration behavior. Potentially the most powerful model for predicting reaction rates and

microstructure development, HydratiCA is also the most complex and computationally intensive. The NIST researchers have managed important breakthroughs that make HydratiCA more robust and far more efficient. In addition to integrating the experimental findings generated by the UCSB and OSU research teams, the NIST team has parallelized the HydratiCA model to run on multiple central processing units at once and optimize communications among processors, resulting in an efficiency boost of 40 to 200 times. This allows HydratiCA to model cement hydration in real time: simulating 24 hours of hydration now takes 24 hours of run time, rather than several weeks or months.

Case Study: New Models to Predict Concrete Performance (Cont.)

In addition to enhancements to HydratiCA, the NIST team incorporated experimental results into the VCCTL (virtual cement and concrete testing laboratory) website and THAMES (thermodynamic hydration and microstructure evolution simulator). VCCTL has a user-friendly interface, which is connected to a materials database that allows a user to model a 3D microstructure of a cement mix and the hydration of that mix. THAMES builds on the capabilities of VCCTL by allowing small amounts of other materials to be added to the microstructure and determining the impact of these changes on hydration. Unlike VCCTL, THAMES is built with underlying thermodynamics data, which allows for greater accuracy in modeling hydration.

Researchers at Princeton University developed a computer program called SimBNG that models boundary nucleation and growth (BNG) of C-S-H, the cement hydration product that is primarily responsible for strength in cured concrete. A key goal for SimBNG development in this project was to apply as many external constraints on the model as possible, while maintaining quick run times and accurate results. The group at Princeton developed a method for supercritical drying of cement paste that preserves the delicate microstructure of the material without the artifacts that usually result from the capillary pressure exerted during conventional drying.

Figure 6. Photo: Laboratory research methods include dynamic modulus of concrete with the measurement of fundamental resonant frequency. Source: FHWA.



Conclusion

The EAR Program's "Mechanisms of Hydration and Setting of Ordinary Portland Cement in Simple and Complex Systems" project has generated an extraordinary new understanding of the critical processes involved in cement hydration. The range of accomplishments includes sensitive and innovative analytical methods and a deepened understanding of fundamental chemical processes governing hydration; with this understanding, practitioners can plan concrete projects that use resources more efficiently and produce structures that are stronger and more durable.





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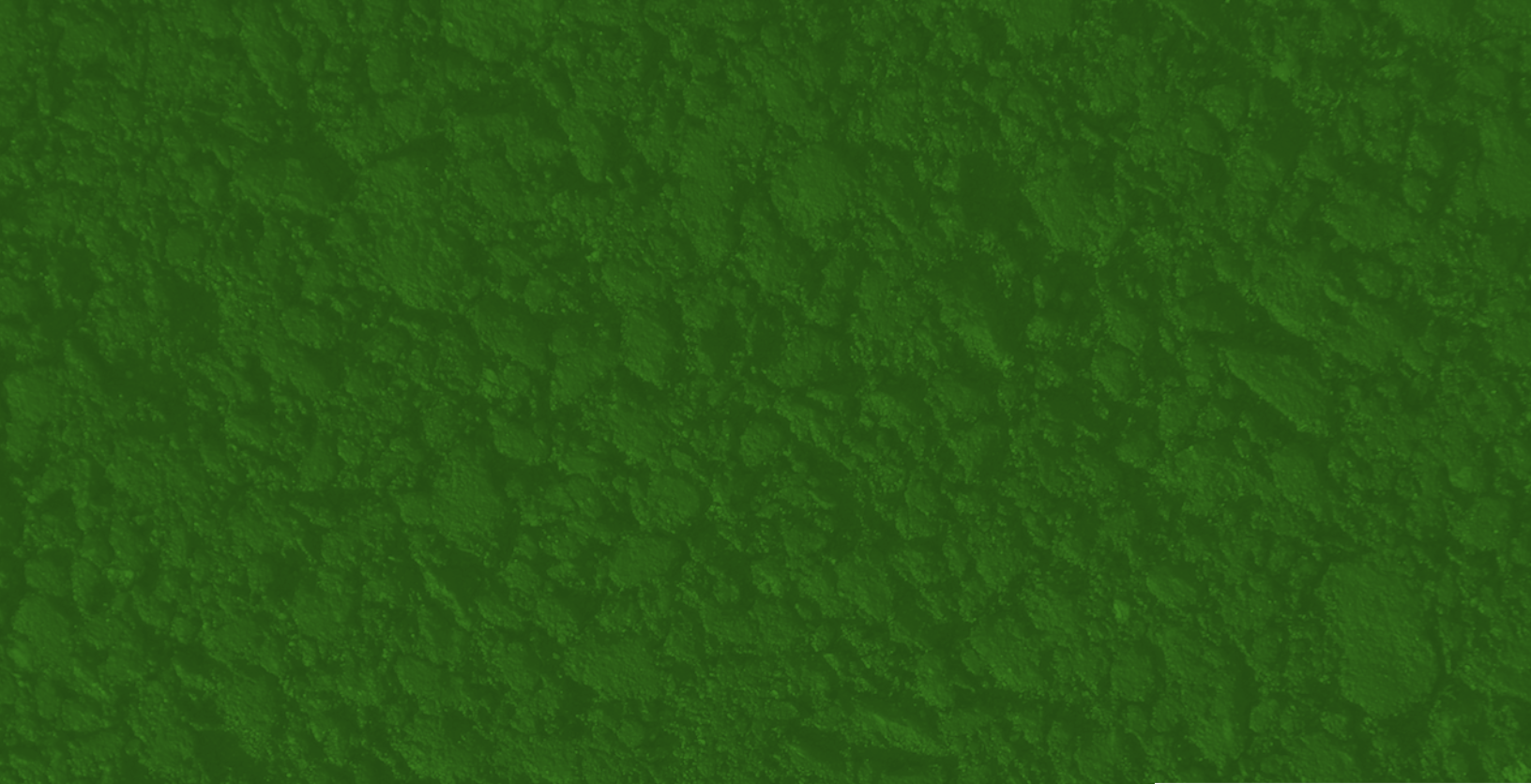


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