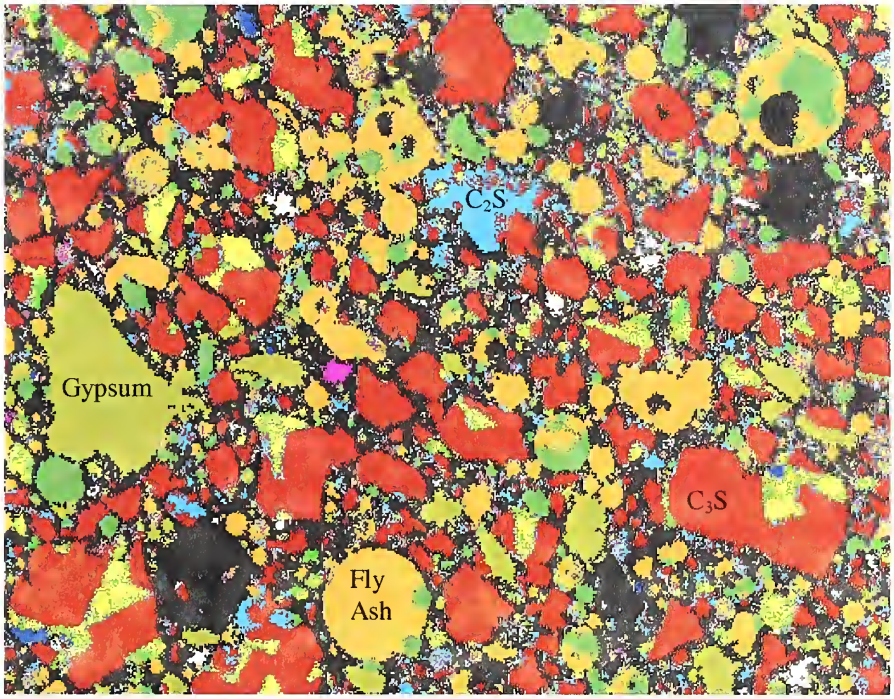
NISTIR 6840

The Virtual Cement and Concrete Testing Laboratory Consortium Annual Report 2001

Editor: Dale P. Bentz



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Editor: Dale P. Bentz
Building and Fire Research Laboratory

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U.S. Department of Commerce
Donald L. Evans, Secretary

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Phillip J. Bond, Under Secretary for Technology

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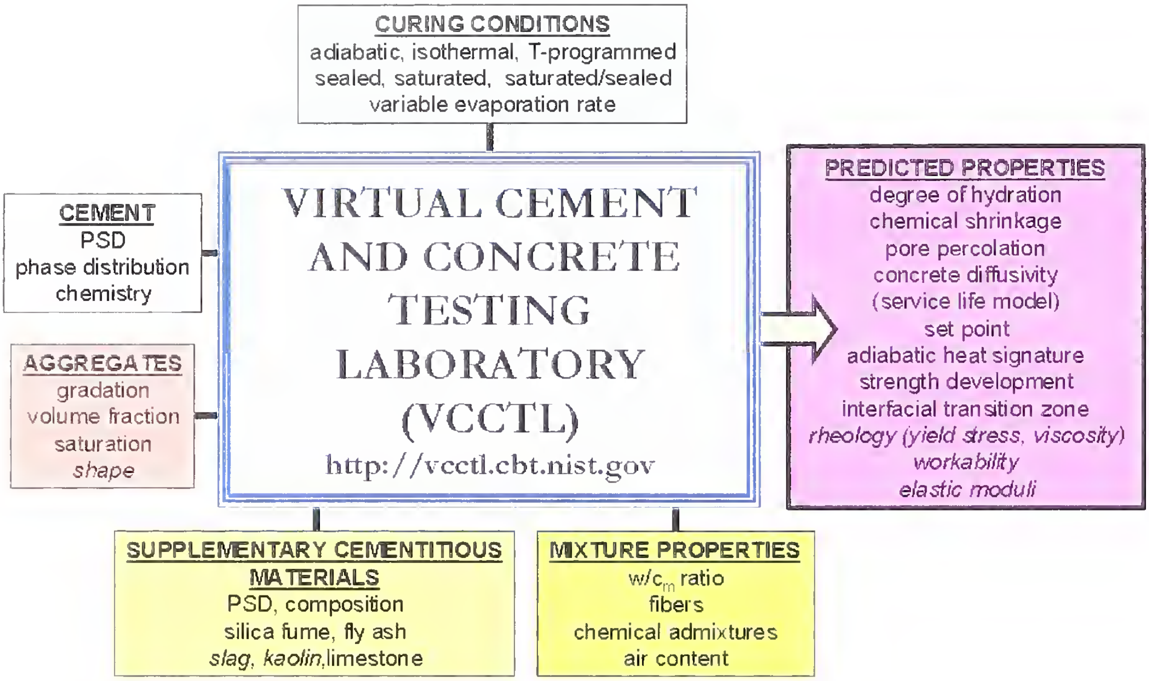
Arden L. Bement, Jr., Director

OVERVIEW

The Virtual Cement and Concrete Testing Laboratory (VCCTL) consortium was formed in January 2001. Headquartered in the Building and Fire Research Laboratory (BFRL) at the National Institute of Standards and Technology (NIST), the consortium currently consists of three NIST laboratories: BFRL, the Information Technology Laboratory (ITL), and the Materials Science and Engineering Laboratory (MSEL), and six industrial members: Cemex, Dyckerhoff Zement GmbH, Holcim Inc., Master Builders Technologies (MBT), the Portland Cement Association (PCA), and W.R. Grace & Co.- Conn. The overall goals of the consortium are to develop a virtual testing system to reduce the amount of physical concrete testing and to expedite the research and development process. This will result in substantial time and cost savings to the concrete construction industry as a whole.

The consortium is governed by the VCCTL consortium oversight board, consisting of one representative from each of the six companies and the VCCTL consortium manager from NIST. The oversight board meets two to three times per year to review research progress and set the scope and agenda of future research. Within each industrial participant's laboratory, one researcher is assigned to participate in the research programs of the consortium. Once per year, the most recent version of the VCCTL system software is installed on a Linux-based PC at each participating member's laboratory. Thus, during 2001, version 1.0 of the VCCTL software was installed at each participating laboratory. A diagram overviewing the VCCTL system is provided in the figure below.

While the ultimate goal of the consortium is to address durability and service life prediction, it was recognized that initial efforts must be concentrated towards enhancing the current microstructure models and property calculations. With this in mind, the three major initial research topics of the consortium are: 1) enhancements to the cement hydration and microstructure development model to consider additional supplementary materials such as slags and limestones and the prediction of pore solution concentrations, 2) computation of the elastic properties (elastic modulus, creep, shrinkage) of three-dimensional microstructures, and 3) experimental measurement and computer modeling of the rheological properties (viscosity, yield stress) of cement-based materials. Progress made during the first year in each of these three areas will be presented in this report.



Caption: Diagram showing inputs and outputs of the Virtual Cement and Concrete Testing Laboratory system. Items listed in italics indicate topics currently being pursued in the VCCTL consortium that ultimately will be included in the system.

IMPORTANCE AND RELEVANCE OF THE VCCTL

Potential Advantages of Computer Modeling for the Cement Industry

(Dr. Claus-Jochen Haecker, Dyckerhoff Zement GmbH, Wiesbaden, GERMANY)

Cement is a rather old building material. In the massive masonry constructions and brickworks of the Romans, we find our present-day method of uniting blocks and slabs of stone with mortar, consisting of a mixture of sand and hydraulic binder. Portland cement, as we know it today, is not quite this old, but it has been used for at least 150 years. Although cement is such an old and well-accepted system, we are still far from a full understanding of what happens during cement hydration and how its physical properties develop during the hydration process. The lack of understanding is only partially due to not knowing the system. There is a lot of empirical knowledge compiled for cementitious systems. Cementitious systems are very complex and simple empirical approaches to describe these systems usually fail. In order to be able to realistically describe cementitious and/or concrete systems, models that take into account all relevant parameters are needed. Due to the complexity of the systems to be described, these models must be computer-based.

Today, new and better cements are still developed empirically. Empirical studies require a great deal of experimental work and thus are very time consuming and expensive. Furthermore, quality control in a cement plant requires extensive testing. Computer modeling offers the advantage of replacing, or at least reducing, time-consuming and expensive experimental work by fast and inexpensive computer simulations. These advantages may be useful in the following areas:

1. Cement manufacturers are motivated to satisfy their customers and thus often have to adjust the performance of their cements to meet specific needs of their customers. Optimizing cement performance implies experimental studies, some of which can be done in a laboratory, and some of which must be done in the production plant and thus interferes with the production process. Potential means to optimize cements include for example:
   - changing the clinker chemistry,
   - adjusting the sulfate source, or
   - adjusting the cement particle size distribution.

   Reliable results from computer simulations can help to reduce expensive studies in production plants.
2. In order to assure cement quality, every cement is usually tested after 2 d, 7 d, and 28 d. Under detrimental circumstances, quality problems may be evident after 28 d. It might happen that after 28 d the corresponding cement is already in place and thus quality assurance measurements arrive too late. Through computer simulations, quality problems are evident after a couple of hours of computer time, while there is still enough time to take corrective measures.
3. In the course of quality assurance according to European Standard 197, a cement producer has to do the following (in general, in addition to further physical properties to be tested):
   - For each produced cement take two samples every week

- Test compressive strength after 2 d, 7 d, and 28 d.

By reliable computer simulations, the cement industry in Europe could eliminate 312 compressive strength tests per year for each cement produced and thus could considerably lower production costs.

4. Research and development processes can be accelerated. When a new cement is created, information about its durability is required. If durability is tested physically, it may take a couple of years to obtain reliable data. If durability could be simulated, these results would be available after a couple of hours/days and thus R & D would be accelerated to a great extent.

Modeling and Simulation – Its Value to the Chemical Admixture Industry

(Dr. Davide Zampini, Master Builders Technologies, Zurich, SWITZERLAND)

Specifications in the construction industry are becoming more demanding and the applications more sophisticated. Thus, concrete structures are required to fulfill rheological, mechanical, and durability performances that are well above standards established in the past. Consequently, the need to satisfy performance requirements in the construction industry has led to the development of new chemical admixtures and different types of cements.

However, one of the major problems encountered in field applications is the lack of an in-depth understanding of interactions between cements and chemical admixtures. Due to the complex nature of cement chemistry, the variety of cements available, and the different types of chemical admixtures, it is extremely difficult to gain proper insight into the fundamentals underlying cement-admixture interactions. This occasionally leads to unforeseen or unexpected behavior of concrete in field applications. Therefore, there is still a lot of research needed in the area of cement-admixture interactions.

Modeling and simulation tools like the VCCTL provide a means to gain understanding of cement-admixture interactions by allowing researchers to evaluate, in very short time spans, a large number of possible hydration reactions. In its current state, the VCCTL is still limited regarding the possibilities of introducing or varying the possible chemical effects resulting from the presence of an admixture in a hydrating cementitious system. Nonetheless, the expertise and guidance provided by NIST scientists to the consortium members is impeccable. Thus, through proper consulting, chemical admixture companies participating in the VCCTL consortium will be in a position to master the VCCTL by introducing code modifications that will enable the modeling and simulation of cement-admixture interactions and predict their influence on rheological properties and hydration.

CEMENT HYDRATION

"Cement paste is the glue that holds concrete together and the sponge through which aggressive agents enter. Understanding its microstructural development during hydration and degradation during exposure are critical to predicting concrete properties and field performance."

Background

Research on the NIST pixel-based three-dimensional cement hydration and microstructure development (**CEMHYD3D**) model commenced in 1989. Currently recognized as the most extensive, complete, and robust cement microstructure model in the world, CEMHYD3D [1,2] serves as the core of the Virtual Cement and Concrete Testing Laboratory software [3]. Starting from a measured particle size distribution (PSD) of the cement and a detailed SEM/X-ray image set for the cement powder, a starting three-dimensional microstructure of cement (calcium sulfate, fly ash, etc.) particles in water is created. The four major clinker phases (C_3S, C_2S, C_3A, and C_4AF) are then distributed amongst the three-dimensional "cement" particles to match the phase volume fractions, surface fractions, and correlation structures measured on the real 2-D cement powder image set. This microstructure is then submitted to the cement hydration model where the user may specify curing conditions in terms of both heat (isothermal, semi-adiabatic/adiabatic, temperature-defined) and moisture (saturated, sealed) transfer conditions. As hydration proceeds in the model, a set of output files is created to monitor specific properties as a function of hydration time or achieved degree of hydration. Properties monitored include chemical shrinkage, heat release, setting, capillary porosity percolation, pH and pore solution concentrations, and strength development.

Progress in 2001

During 2001, research focused on three subtopics: 1) modeling and experimental measurements on systems containing limestone fillers, 2) incorporation of the reactions for slag into CEMHYD3D, and 3) modeling of the pH and ion concentration of the pore solution during hydration. Substantial progress was made on each of these topics as will be detailed below.

Limestone additions to cement are commonplace throughout the world, with the exception of the U.S. In general, small (<= 15 %) additions of limestone have little or no detrimental effects on cement properties and result in a cost savings for the cement producers. Additionally, in low w/c ratio concretes, there is insufficient space for all of the cement to hydrate completely, so that some of the cement may be acting as a rather expensive reinforcing filler. Within the VCCTL consortium, we have been exploring the concept of replacing the coarsest cement particles by limestone fillers in low w/c ratio (<=0.3) concretes. To do this, we have carried out both computer modeling and experimental research during 2001. From the modeling side, reactions for limestone were added to the CEMHYD3D software. The limestone reacts with aluminates to form a calcium aluminate monocarbonate (Afmc) reaction product, reducing the monosulfoaluminate (Afm) content in the hydrated system [4]. From an experimental viewpoint, the consortium has examined the concept of replacing the coarsest fraction of the cement particles by limestone filler of a similar size. At the suggestion of **Dyckerhoff**, OMYA Inc. was contacted and a high purity limestone with a median particle size of about 30 μm was obtained[1]. The limestone and Cement and Concrete Reference Laboratory (CCRL) Cement 135 were classified at the facilities of **W. R. Grace** in Cambridge, MA in July, 2001, separating both materials at a cutoff size of 30 μm. The achieved particle size distributions for the separated materials were evaluated by **Dyckerhoff**. The coarse limestone particles were then blended with the fine cement particles at NIST using a V-blender. High performance cement mortars (w/s=0.3) with and without 15 % limestone replacement of the coarse cement particles were prepared and the compressive strength of mortar cubes evaluated after 7 d and 56 d of curing in limewater. After 7 d, the system with 15 % limestone had a 10 % lower compressive strength than the "control" system. But after 56 d of curing, both systems achieved compressive strengths of 99 MPa, demonstrating the viability of replacing coarser cement particles by limestone in low w/c ratio mortars and concretes.

A set of hydration reactions for slag has also been incorporated into version 2.0 of the VCCTL (CEMHYD3D) software. To use this option, the user must create a slag characteristics file for each slag being considered. To appropriately model a given slag, the user should obtain its particle size distribution and oxide composition. The hydration reactions for slag have been developed and partially validated based on materials received from both **Holcim** and **Dyckerhoff**. The slag reacts with a fraction of the calcium hydroxide produced from the cement hydration to produce a "slag"-gel hydration product (containing calcium, silicon, aluminum, magnesium, and possibly sulfate). If the slag contains excess aluminum relative to its magnesium content (with regard to the formation of a hydrotalcite-type phase), the excess aluminate participates in reactions with the calcium sulfate in the cement to form ettringite and monosulfoaluminate (and hydrogarnet when sulfate is not readily available). Validation of these reactions has been based on measurements of chemical shrinkage of blended cements (typically 30 % slag, 70 % cement), measurements of calcium hydroxide production/consumption, and analysis of hydrated microstructures via X-ray diffraction and scanning electron microscopy. To date, good agreement between model predictions and experimental measures has been obtained.

Based on the approach developed and published by Prof. Hal Taylor [5], computation of the alkali (potassium and sodium) concentration in the pore solution during hydration has been added to the CEMHYD3D software. The user must supply an alkali characteristics file that contains information on the total and readily (1 h) soluble alkali contents of the cement of interest. The pH prediction module of CEMHYD3D computes the pH of the pore solution along with the concentrations of the following ions:

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[1] Certain commercial equipment, instruments, or materials are identified in this report. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

calcium, potassium, sodium, hydroxide, and sulfate. At early times, while gypsum remains present in the hydrating microstructure, ion concentrations are computed based on the maintenance of equilibrium between the pore solution and the solid phases of calcium hydroxide and gypsum, along with an electroneutrality condition. At later ages, the sulfate concentration is assumed to go to zero and only the equilibrium with calcium hydroxide is maintained. Based on experimental data obtained at NIST (using a pore press and ion chromatograph) and Laval University, there is good agreement between model predictions and experimental measurements for both pore solution pH and the concentration of [K+] and [Na+] in solution. Ultimately, the pore solution concentration and pH will be linked to the reactivity of the cement clinker phases and mineral admixtures such as slag and fly ash within the CEMHYD3D model.

In addition to the research subtopics described above, at the suggestion of consortium members, several enhancements to the CEMHYD3D model were made during 2001. These include the incorporation of semi-adiabatic curing conditions, the modeling of flash setting via rapid hydration of the aluminate phases, and the development of a set of supplementary computer programs for modeling the hydration/drying of a 2 mm thick (100 pixels by 100 pixels by 2000 pixels) paste specimen. All of these enhancements will be distributed to the members as part of version 2.0 of the VCCTL software.

Future

The CEMHYD3D components of the VCCTL will continue to evolve during the lifetime of the consortium. As new materials are proposed or evaluated for use in cement-based systems, their reactions and interactions will be incorporated into the hydration model. The slag reactions will be further validated by **Cemex** and **Dyckerhoff,** once version 2.0 of the VCCTL has been installed in their laboratories. Likewise, research on establishing a linkage between pore solution concentrations, pH, and hydration rates will be conducted in a collaborative effort between **Cemex, Dyckerhoff**, and NIST, based on a controlled set of experiments and modeling using version 2.0 of the VCCTL. Installation of version 2.0 of the VCCTL at each member laboratory is planned for February 2002.

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ELASTIC PROPERTIES

"In terms of microstructure and number of individual phases, cement paste is probably the most elastically complex random composite material routinely used by humankind, and yet it has probably had the least attention paid to it by composite theoreticians and experimentalists."

Background

Almost all real materials are multi-phase materials, whether deliberately, when formulating a composite; inadvertently, by introducing impurities into a nominally mono-phase material; or by the very nature of the material components, as in the case of cement-based materials. In any event, predicting the elastic properties of such a material is absolutely dependent on two types of information for each phase: how each phase is arranged in the microstructure, and the elastic moduli of each phase. Cement paste is extraordinarily complex elastically, with many (20+) different chemically and elastically distinct phases and a complex microstructure. This complexity further increases in concrete, as aggregates are added.

A finite element package for computing the elastic moduli of composite materials has been written at NIST and has been available for several years [1]. The basic FORTRAN program is called elas3d.f. The program takes a 3-D digital image of a microstructure, assigns elastic moduli tensors to each pixel according to what material phase is present, and then computes the effective composite linear elastic moduli of the material. This program is highly optimized, and has worked successfully in many different material microstructures, including ceramics [2], metal alloys [3], closed and open cell foams [4-6], gypsum plaster [7], oil-bearing rocks [8], and 2-D images of damaged concrete [9]. It can take as input the cement paste microstructure as given by the CEMHYD3D model, and operate on it to compute the cement paste elastic moduli. However, the elastic moduli output is only as good as the phase elastic moduli input. Portland cement paste has many different phases. One goal of the VCCTL consortium is to measure the individual phase elastic moduli needed, along with cement paste elastic moduli as a function of degree of hydration, cement particle size, and w/c ratio, to develop elas3d.f into a tool that is an accurate and easy-to-use component of the VCCTL software.

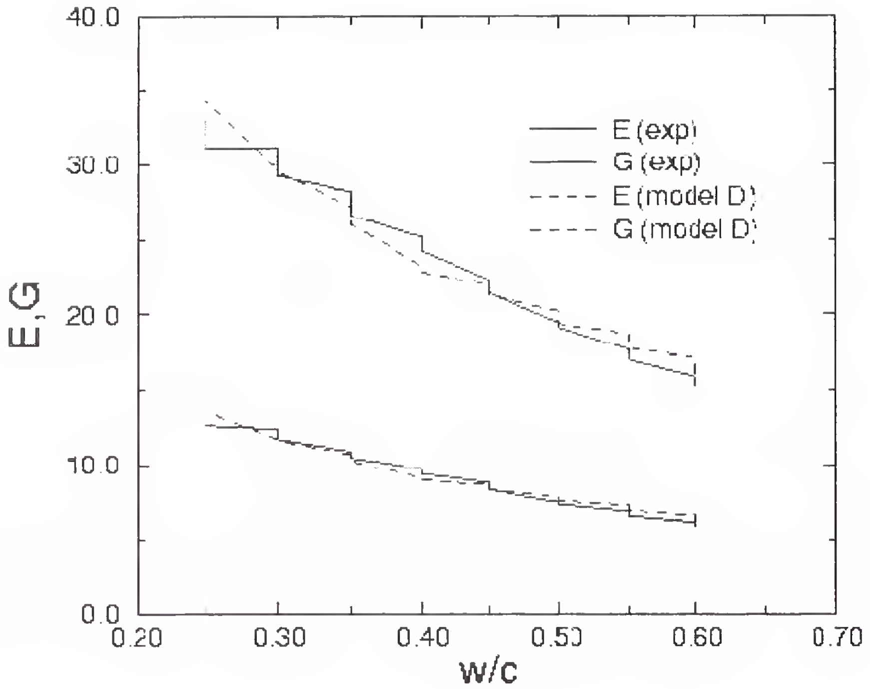
Progress in 2001

During 2001, **Dyckerhoff** measured the elastic properties of a series of cement pastes of different w/c ratios (between 0.25 and 0.60) after 28 d and 56 d of saturated curing, along with some assorted early-age data. They are also gearing up to measure individual phase elastic moduli. NIST researchers have combed the geological literature to find the available cement/cement paste mineral elastic moduli data. There have also been some new data reported in the cement literature on cement clinker minerals and on C-S-H. Using all this available individual phase elastic moduli data, NIST has modeled the hydration of these cements (in each case, matching the degrees of hydration achieved experimentally, as determined using the procedure documented in VCCTL Technical Note 01) and then computed the elastic properties. Agreement between model and experiment is quite reasonable for these later age specimens. The figure below shows the 28 d and 56 d data, plotted against w/c ratio, for both experimental and model results. The agreement is usually within 5 %, although some points only agree within 10 %. Previous later age experimental results generated at **Dyckerhoff** for cements of different finenesses are also well predicted by the model; at equivalent degrees of hydration, fineness did not have a significant effect on elastic moduli.

This success has led to the incorporation of a C version of the elas3d program (called **cpelas.c)** into version 2.0 of the VCCTL package. After generating a cement paste microstructure with a given cement, at a specified w/c ratio and hydrating to a given degree of hydration, the user will be able to compute the elastic moduli of the generated cement paste. The hydrated microstructure could also be exposed to leaching conditions (removing CH) and the elastic moduli of the "degraded" cement paste computed. It should be remembered that at this time only later age predictions (above about 45 % hydration) are currently valid using the elastic codes. Early-age predictions will be worked on in the future, and are described below.

Future

Future efforts will focus on refining the elastic property values of the "pure" phases present in the model (particularly ettringite and C-S-H) and extending the predictions to early age specimens and to concrete. In the near future, the elastic computer codes will be parallelized to provide faster turnaround and the possibility to quickly run larger higher-resolution microstructures. Early age predictions of elastic moduli are more difficult, because of the low connectivity between phases at early ages. The higher resolution available on the parallel machines should help resolve this problem, as well as alterations in the finite element basis of elas3d.f. It is also true that cement paste, especially at early ages, is a viscoelastic material. Incorporating these properties into the elastic moduli code is also an item for future work. Finally, it is important to use accurate elastic moduli predictions of cement paste, the matrix of concrete, to accurately predict concrete elastic moduli and thereby predict concrete strength. Effective medium theories for taking cement paste results and predicting concrete results are available [10], and will be further developed in future work.



Caption: Showing how well the elastic moduli predictions (E- Young's modulus, G-shear modulus, both in GPa) compare to experimental data, vs. w/c ratio, for 28 d and 56 d specimens. At each w/c ratio, the upper point is the 56 d value and the lower point is the 28 d value.

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RHEOLOGICAL PROPERTIES

"All the desired hardened properties of concrete cannot be realized in practice, if the material is not properly placed and consolidated. Therefore, the correct prediction of the rheological properties of the concrete are paramount for producing a durable high performance material."

Background

At NIST, an ongoing program exists with the goal of predicting the rheological performance of the concrete from its composition. The prediction is based on several steps: 1) measuring the rheological properties of the cement paste to determine the influence of chemical admixtures and supplementary cementitious materials; 2) mortar rheological properties measurements to determine the influences of air and sand contents; and 3) predicting the concrete properties through a computer simulation [1,2,3] from the measurements on mortar and/or cement paste.

This approach is justified by the fact that most chemical admixtures and supplementary cementitious materials will mainly affect the rheological properties of the cement paste. Mortar measurements provide a representative and stable amount of air, as well as a wide range of sand size distributions and shapes. The progression from mortar to concrete entails the addition of the coarse aggregates. A novel computer simulation approach allows us to take the rheological properties measured on mortar, add the coarse aggregates with their shape and granulometry, and predict the concrete rheological properties. The advantage of this approach is that several tests could be done by one operator on cement paste or mortar in a day, while concrete testing requires several operators and much more material.

This approach obviously needs to be validated at all stages. The rheological properties of cement pastes containing different supplementary cementitious materials (SCM) and high range water-reducing admixtures (HRWRA) were measured and the results were compared to concrete data [4]. The results showed that the rank of the cement paste mixtures was found to match the concrete performance characterized by the conventional slump test. A methodology to measure the rheology of mortars was developed and tests are underway with ideal aggregates (spherical glass beads) in mortar and concrete to validate the model (co-sponsored by the Center for Advanced Cement-Based Materials).

The cement paste and mortar measurements are performed using a parallel plate rheometer with different size plates depending on whether cement paste or mortar is to be measured. The materials are mixed using a high shear mixer with controlled speed and temperature, based on **PCA** methodology [5], to ensure that the material tested experiences conditions typically found in concrete.

The model is based on dissipative particle dynamics (DPD) theory [6,7,8]. This model combines the concepts of cellular automata and molecular dynamics. The original

DPD algorithm uses properties such as conservation of mass and momentum, and Galilean invariance to build a set of equations that track, at all times, the positions of the particles. These particles may represent an agglomeration of molecules or a finite volume of fluid. Modifications of the model allowed improved precision of the numerical calculations. An algorithm modeling the movement of solid objects of arbitrary shape was developed by Koelman and Hoogerbrugge (KH) [7, 8]. A rigid body is represented by "freezing together" a group of particles in the volume occupied by the object. Their position is determined at all times by the Euler equations. The KH method is being modified by Martys at NIST for application to the flow of concrete. The shape of real aggregates can easily be described by this method. Therefore, it will be possible, knowing the medium (mortar) rheological properties, to "virtually" add the aggregates with their shape and size distribution. This will allow the simulation of the rheological properties of a concrete of known composition.

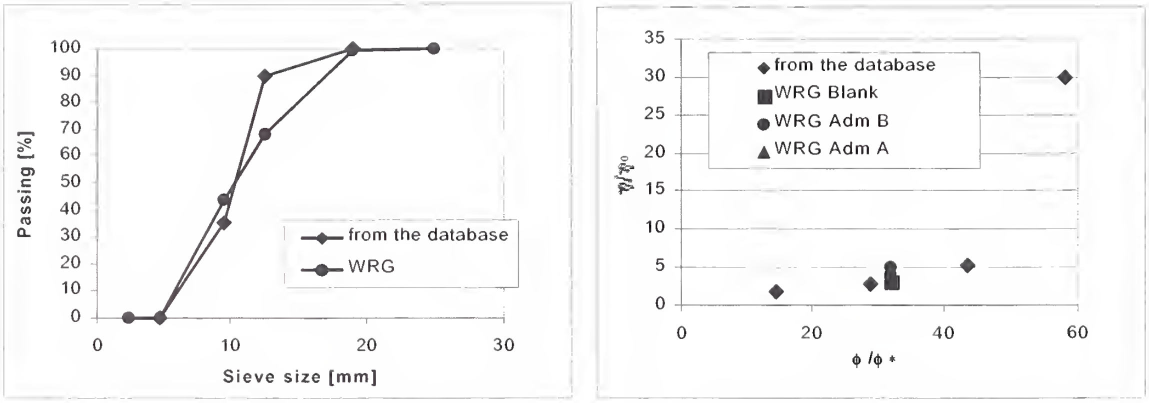
As the simulation requires powerful computers, NIST is building a database that will contain the viscosity of a concrete with a coarse aggregate defined by its shape and size distribution for various aggregate contents. The VCCTL user would search the database for aggregates similar in shape and size distribution to the one being used, measure the mortar properties, and the database will provide the viscosity of the concrete. Obviously, this will allow the reduction of time-consuming and expensive concrete testing by guiding the VCCTL user to the correct concrete composition.

Progress in 2001

During 2001, the methodology to test mortar was developed by modification of the parallel plate rheometer used previously for cement paste. A database was created to collect aggregate size distributions and graphs of relative viscosity versus aggregate concentration. The relative viscosity is the ratio between the viscosity of the concrete and that of the mortar with the same composition as the mortar component in that concrete.

W.R Grace provided mortar and concrete rheological measurements performed using a BML rheometer [9,10]. This enabled NIST to partially validate the model. The coarse aggregate distribution was matched with a distribution in the database and the measured viscosities were plotted on the graph obtained using the model and stored in the database. The figure below shows the results obtained. The agreement is very good. More results of this kind will be used to validate the model. This database will be included in version 2.0 of the VCCTL software.

As most concrete contains entrained air, the DPD model was modified to allow for two fluid phases, i.e., air and paste. Air could be simulated by one or several DPD particles allowing a size distribution to be considered. It is assumed that the entrained air is initially composed of spherical bubbles, which may deform under shear conditions. The two phases repel each other avoiding the dissolution of the air into the paste. This system needs to be validated and further improved as described below.



Caption: Example from the database including the measurements by W.R. Grace (WRG)

Future

Work in the year 2002 will focus on the following topics:

- Evaluation of the influence of air content on the rheological properties of mortar. Air entraining admixtures have been provided by **W.R. Grace** and **MBT**. Several dosages and air contents will be tested and the relation between air content and rheological properties will be examined.
- Continue the collaboration with ACBM to validate both the experimental and simulation techniques by measuring the rheological properties of cement paste, mortar, and concrete. In the latter two materials, the sand and coarse aggregates will be replaced by glass beads or spherical aggregates. Their shape is simple (compared to a crushed aggregate) and therefore the results should be closely simulated by the DPD model.
- The DPD model will be further improved by adding rigid bodies to the two-fluid algorithm to simulate the rheological properties of air-entrained concrete.

The results obtained from this work will be shared with the VCCTL members by preparing a VCCTL Technical Note on the methodology used to measure cement paste and mortar rheology using the parallel plate rheometer. The database incorporated in version 3.0 of the VCCTL package will contain the influence of air entrainment on the relative viscosity.

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[9] Ferraris, C., and Brower, L., editors, "Comparison of concrete rheometers: International tests at LCPC (Nantes, France) in October 2000", NISTIR **6819**, U.S. Department of Commerce, September 2001.
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MATERIALS CHARACTERIZATION

"To avoid 'garbage in, garbage out', an accurate and detailed characterization of the starting (cement) powders is paramount to successfully modeling hydration and performance."

Particle Size Distribution

While the particle size distributions (PSDs) of cements and mineral admixtures are critical to the properties and performance of cement-based materials (as illustrated in the case studies given below), a universally recognized standard method for characterization of the size of cement particles does not currently exist [1,2]. Consequently, the implementation of different measurement methods varies widely within the industry. In general, round-robin tests conducted to date have demonstrated a high variability among results, even between participants using instruments based on the same underlying principles. Potential sources of variability in sizing methods include adjustable instrument parameters or material property data required as input parameters, and fundamental differences due to the nature of the technique itself. In the latter case, it must be acknowledged that different methods may "sense" a different aspect of the size distribution. For instance, a given method may be sensitive to either particle mass, particle number, or projected surface area. As a result, for a polydisperse system, each method produces a distribution with slightly different weighting. Thus, the "mean" particle diameter values are expected to differ.

ASTM Subcommittee C01.25 (Dr. Ferraris of NIST is an active member) is currently discussing the development of a standard test for cement PSD. Its approach consists of two steps: 1) define a consensus PSD curve using a standard cement such as NIST Standard Reference Material 114P, and 2) develop a standard test.

The first step to achieve these goals was to organize round-robin tests. Therefore, in 2001, two round-robin tests were organized, one within ASTM and the other among the participants of the VCCTL consortium. The results were analyzed and the following conclusions drawn:

- The most commonly used techniques for characterization of PSD in cement are as follows:
    1. Laser Diffraction
        a. with the specimen dispersed in liquid (suspension-based)
        b. with the specimen dispersed in air (aerosol-based)
    2. Electrical Zone Sensing (Coulter principle)
    3. Scanning Electron Microscopy
    4. Sedimentation
    5. Sieving

The vast majority of the laboratories (and all the participants within the VCCTL consortium) use the laser diffraction technique, either wet or dry. The wet technique is currently more popular than the dry technique.

- The parameters used by the various laboratories are not the same and the influence of these parameters is not always clearly understood. These parameters range from the properties of the cement, such as its complex diffraction indices, to the model used to interpret the data measured (Fraunhofer or Mie) and the dispersion method or medium (isopropanol, methanol, or ethanol).
- The scatter of the PSD obtained with the various methods on the same cement could be very wide, even if the reproducibility of the results by the same operator is very good. For example, Figures 1 and 2 show the results obtained with the laser diffraction wet and dry techniques during the round-robin conducted by the VCCTL members for CCRL Cements 141 and 142.

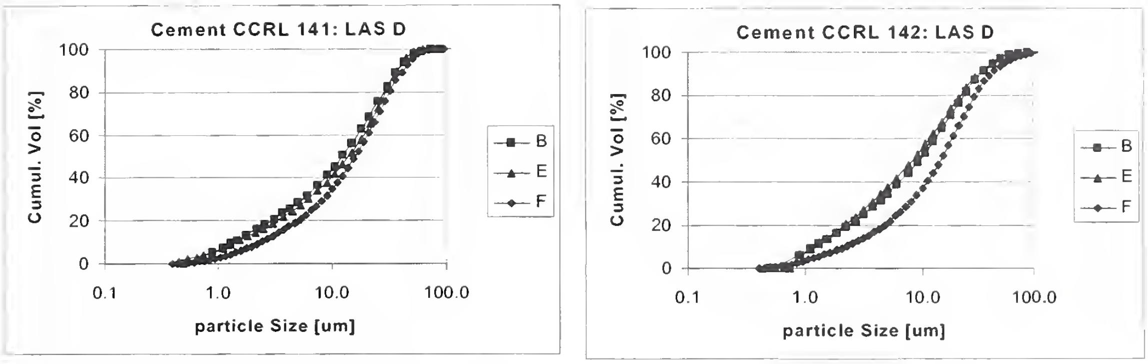


Figure 1: Laser Diffraction, Dry PSD

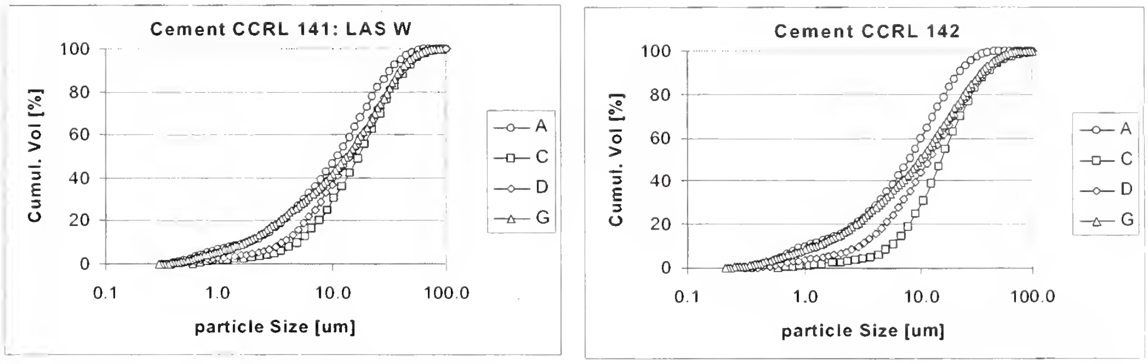


Figure 2: Laser Diffraction, Wet PSD

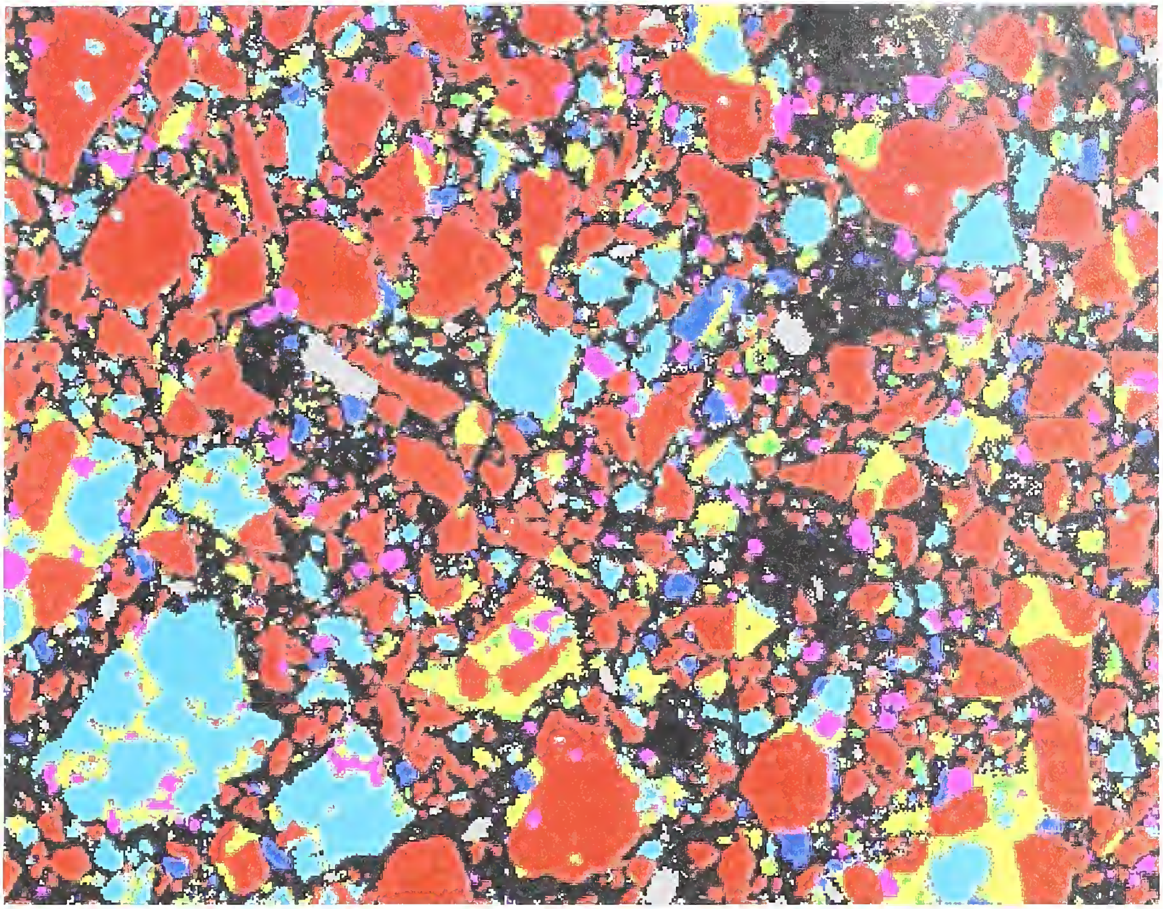
NIST has initiated a study to help the VCCTL and ASTM to develop a standard test for cement PSD determination by laser diffraction. On the one hand, the collaboration with ASTM will yield at first a "consensus" PSD using NIST cement 114P. This cement is normally sold to laboratories to calibrate their Blaine fineness device. The consensus PSD of this cement will be determined by a round-robin test in collaboration with CCRL and ASTM. More than 200 laboratories will be asked to measure the PSD of this cement and a CCRL cement in the context of the existing CCRL proficiency sample program. This collaboration will allow us to determine a consensus PSD that could be used to calibrate the most commonly used particle sizing techniques in the industry.

On the other hand, NIST will examine the influence of various parameters on the results obtained using the laser diffraction wet method. This study will yield the methodology needed to standardize the measurements of cement PSD with this technique. A laser diffraction dry method device will also be acquired by NIST and the same study will be conducted on this instrument.

In conclusion, by the end of 2002, there should exist a much clearer picture of how cement PSDs should be measured using a laser diffraction method. There will also have been developed a "reference" material that can be used to compare the results from different laboratories.

SEM/X-ray Imaging

NIST has pioneered the use of SEM/X-ray imaging for the quantitative characterization of cement powders [3]. This analysis produces a 2-D image of the cement powder in which each pixel is identified as one of the major phases of portland cement as shown below. The resultant image can then be further analyzed to determine phase area fractions, phase surface area (perimeter) fractions, and phase(s) correlation functions. These characteristics are then duplicated in the computer generated three-dimensional microstructure of cement particles in water. Within the VCCTL consortium, a round robin SEM/X-ray analysis of CCRL Cements 141 and 142 is currently underway with participation by NIST, **Dyckerhoff, MBT**, and **W.R. Grace.**



Caption: Processed 2-D image of CCRL Cement 142: red is C_3S, aqua is C_2S, green is C_3A, yellow is C_4AF, grey is calcium sulfate, blue is potassium sulfate, magenta is periclase and white is free lime. Image is 256 μm x 200 μm.

References:

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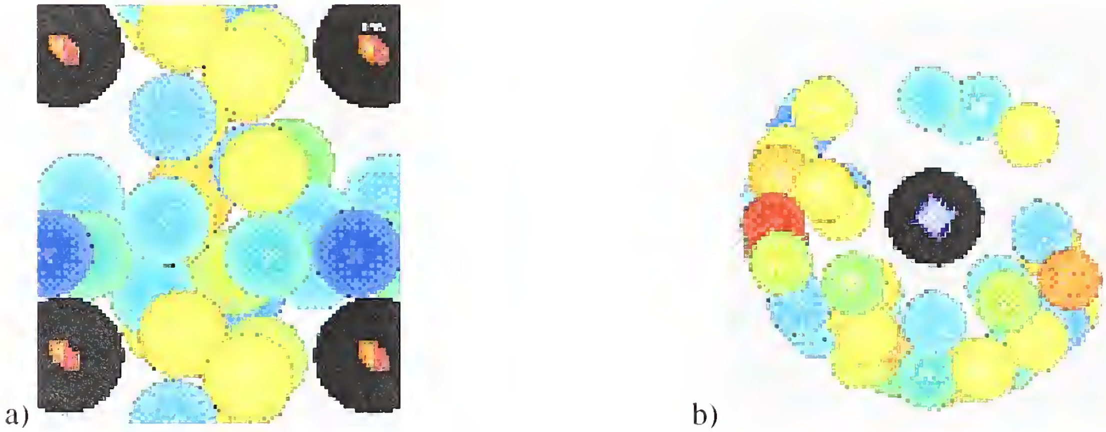
VISUALIZATION

"A picture is worth a thousand words. Immersing oneself in a three-dimensional microstructure is worth even more."

Visualization has always been a critical component of the development of the CEMHYD3D computer model. The initial models (circa 1989) were developed on a PIXAR imaging computer in the NIST laboratories (the same PIXAR that has gone on to create such famous animation movies as *Toy Story* and *A Bug's Life*). Using this machine, the "hydration" could be viewed interactively, greatly expediting algorithm development and debugging activities. As in most areas of computer technology, hardware and software have progressed significantly in the last twelve years. Today, the visualization facilities of the Information Technology Laboratory at NIST continue to be state-of-the-art and heavily utilized in the development of models of cement-based materials' microstructure and performance.

One of the newest additions to the visualization facilities is an Immersive Virtual Reality environment based on a RAVE (Reconfigurable Automatic Virtual Environment) hardware system coupled with DIVERSE software. The RAVE is a two-wall immersive environment with two 2.44 m x 2.44 m (8' x 8') screens flush to the floor oriented 90 degrees to form a corner. DIVERSE handles the details of the stereoscopic projection and the I/O for head and wand tracking. Using the RAVE environment, the researchers may stand within the concrete during flow, or within the cement paste during hydration, to literally "see and feel" what is happening within these complex three-dimensional microstructures. The visualization also plays an important role in the validation of the algorithms and the correctness of complex systems like the flow of fluid concrete within different geometries as shown in the figure below.



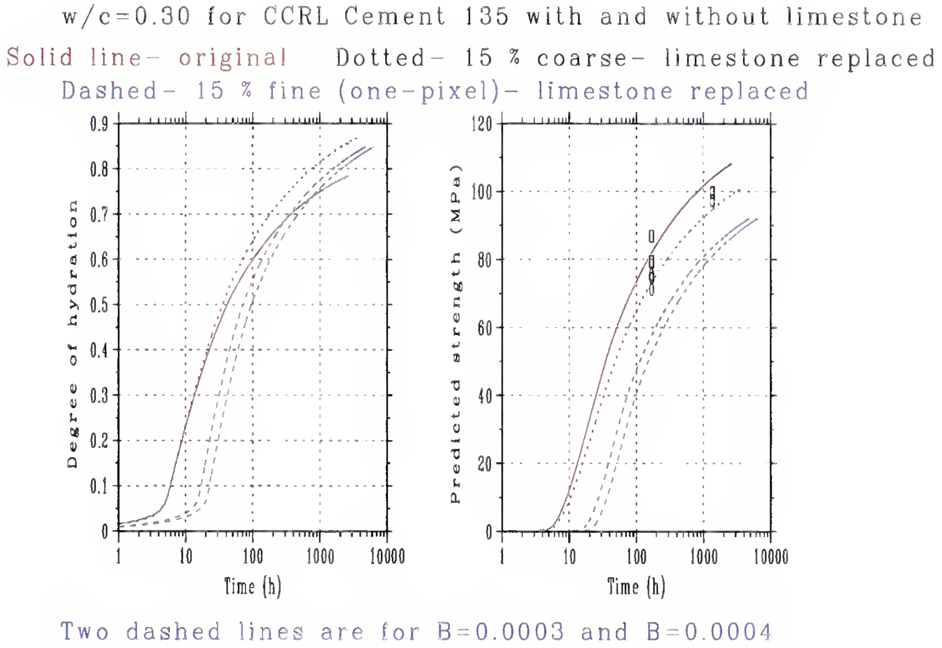
Caption: Static images from simulations of a) flow through a system (grid) of four steel reinforcing bars, and b) flow of a suspension of spheres in a model rheometer with a coaxial geometry. Visualizations are available at http://math.nist.gov/mcsd/savg/vis/concrete **.**

VCCTL CASE STUDIES

I. Replacement of "Coarse" Cement Particles by Limestone Fillers – Hydration and Strength Development

Key Facts:

- Computer simulations suggest that in low w/c ratio concretes, coarser cement particles can be replaced by limestone filler with little reduction in long term compressive strength. A w/c ratio of 0.3 may allow a 15 % replacement by volume, a w/c ratio of 0.25 a 20 % replacement.
- Further simulations (see figure below) suggest that replacing the finest cement particles by limestone (as would be expected when the two materials are interground) will result in a substantial decrease in both early age and longer term compressive strengths.
- Experimental validation of this concept has demonstrated no measurable difference in 56 d compressive strengths for w/s=0.30 with and without 15 % by volume coarse cement particle (>30 µm) replacement by limestone, both systems achieving compressive strengths of 99 MPa at the NIST laboratory.


w/c=0.30 for CCRL Cement 135 with and without limestone

Solid line- original Dotted- 15 % coarse- limestone replaced

Dashed- 15 % fine (one-pixel)- limestone replaced

Two dashed lines are for B=0.0003 and B=0.0004

Caption: **Effects of replacement of either "fine" or "coarse" cement particles by limestone filler on degree of hydration and strength development.**

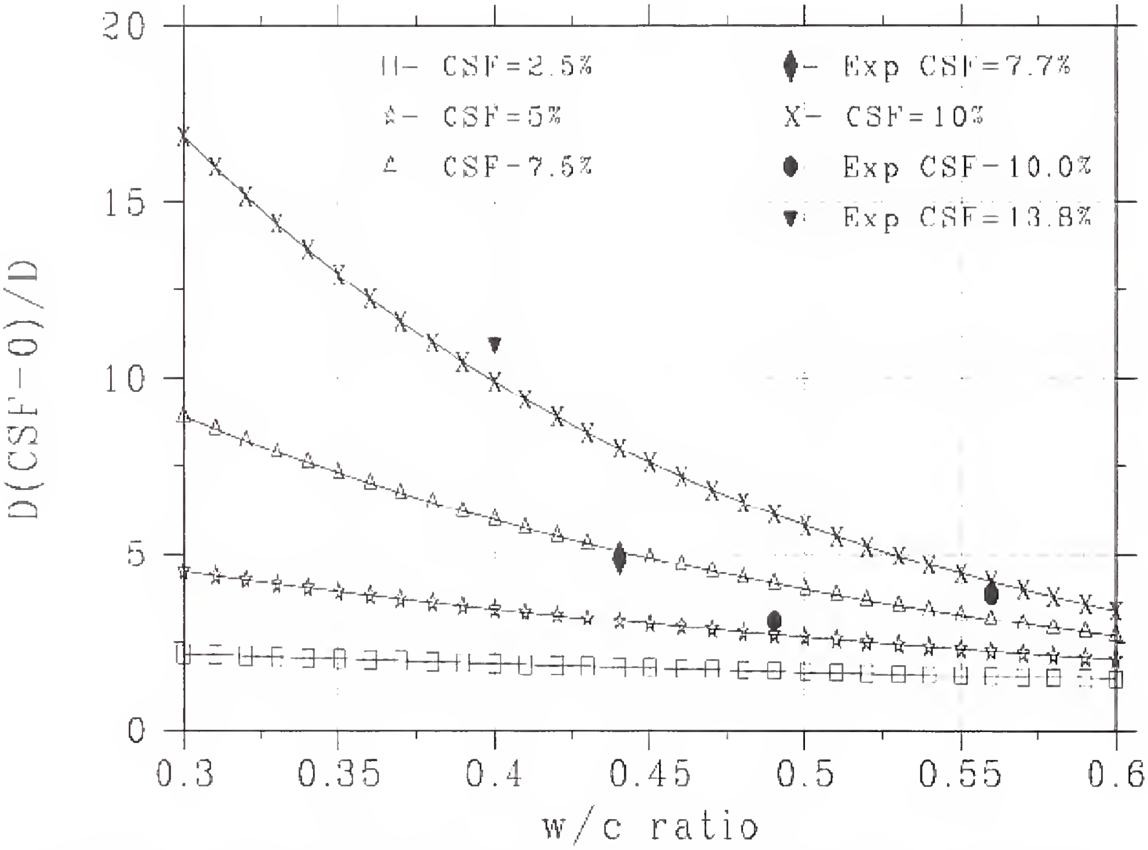
Reference:

Bentz, D.P., and Conway, J.T., "Computer Modeling of the Replacement of "Coarse" Cement Particles by Inert Fillers in Low w/c Ratio Concretes: Hydration and Strength," Cem. Concr. Res., **31** (3), 503-506, 2001, available at http://ciks.cbt.nist.gov/~bentz/cemrepl/cemrepl.html .

II. Multi-Scale Modeling of the Influence of Condensed Silica Fume Additions on the Diffusivities of Cement Pastes and Concretes

Key Facts:

- Silica fume influences concrete diffusivities in three ways: 1) densifying the microstructure of the interfacial transition zones, 2) reducing the overall capillary porosity for a fixed degree of cement hydration, and 3) producing a pozzolanic C-S-H (formed from the reaction between silica fume and calcium hydroxide) that has an inherent diffusivity to chloride ions about 25 times lower than that of the C-S-H gel formed from conventional cement hydration.
- The influence of silica fume on concrete diffusivity is a strong function of w/c ratio (see figure below). For low w/c ratio concretes, silica fume additions on the order of 10 % may reduce the concrete diffusivity by a factor of fifteen.



Caption: Computed multiplicative increase in diffusion resistance due to silica fume addition vs. w/c ratio for an assumed degree of hydration of 0.675. Filled data points are experimental data of Hooton et al. (7.7 % and 13.8 %) and of Alexander and Magee (10 %).

References:

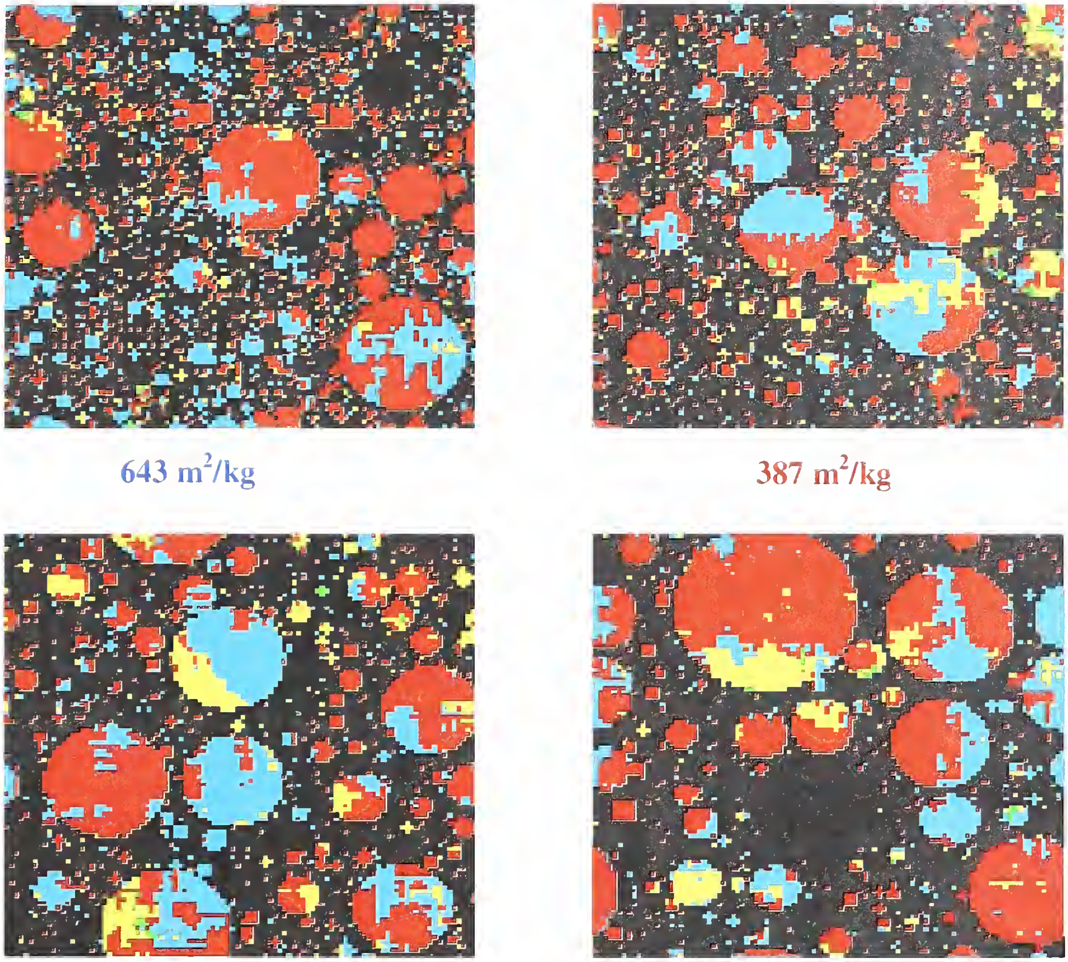
Bentz, D.P., Jensen, O.M., Coats, A.M., and Glasser, F.P., "Influence of Silica Fume on Diffusivity in Cement-Based Materials. I. Experimental and Computer Modeling Studies on Cement Pastes," Cem. Concr. Res., **30**, 953-962, 2000, available at http://ciks.cbt.nist.gov/~bentz/csfdiff/csfdiff.html .

Bentz, D.P., "Influence of Silica Fume on Diffusivity in Cement-Based Materials. II. Multi-Scale Modeling of Concrete Diffusivity," Cem. Concr. Res., **30**, 1121-1129, 2000, available at http://ciks.cbt.nist.gov/~bentz/csfdiff2/csfdiff2.html .

III. Influence of Cement Particle Size Distribution on Properties and Performance

Key Facts:

- Coarser cements significantly reduce early age hydration rates and heat release (and strength development), and thus require that more attention be paid to proper curing.
- At equivalent degrees of hydration, the diffusivities of "fine" and "coarse" cements are similar.
- Coarser cements significantly reduce autogenous shrinkage (and early age cracking).



643 m^2/kg

387 m^2/kg

254 ^{2}/kg

212 m^2/kg

Caption: Two-dimensional images of cement powders of various finenesses (w/c=0.35).

References:

Bentz, D.P., and Haecker, C.J., "An Argument for Using Coarse Cements in High Performance Concretes," Cem. Concr. Res., **29**, 615-618, 1999, at http://ciks.cbt.nist.gov/~bentz/fineness/psdwater.html

Bentz, D.P., Garboczi, E.J., Haecker, C.J., Jensen, O.M., "Effects of Cement Particle Size Distribution on Performance Properties of Cement-Based Materials," Cem. Concr. Res., **29**, 1663-1671, 1999, available at http://ciks.cbt.nist.gov/~garbocz/finetwo/paper2col.html .

Bentz, D.P., Jensen, O.M., Hansen, K.K., Oleson, J.F., Stang, H., and Haecker, C.J., "Influence of Cement Particle Size Distribution on Early Age Autogenous Strains and Stresses in Cement-Based Materials," J. Amer. Ceram. Soc., **84** (1), 129-135, 2001, available at http://ciks.cbt.nist.gov/~bentz/finedtu/.

NIST EQUIPMENT AND FACILITIES

Building Materials Division:
Responsible: Paul Stutzman, Chiara Ferraris, Eric Byrd and Dale Bentz

Microscopy

- **Scanning electron microscope** equipped with solid-state backscattered electron detector. Digital image capture, processing and analysis capabilities. X-ray microanalysis capabilities for qualitative and quantitative spot analyses as well as imaging element spatial distribution.
- **Automated X-ray powder diffractometer** with nine-position sample changer, adjustable optics, diffracted beam monochromater
- **Automated X-ray powder diffractometer** soon to undergo modifications fitting the sample chamber with an environmental cell. The chamber is anticipated to allow control of relative humidity and to exclude CO_2.
- **Light Microscopy Facilities:**
  - Stereo microscope utilizing apochromatic objectives and single optic axis
  - Polarized light petrographic microscope
  - Reflected/transmitted light microscope
- **Atomic force microscope** utilizing an environmental chamber
- **Confocal microscope**
- **Transmission electron microscope** (Analytical Chemistry Division)
- **Specimen preparation facilities** for SEM, optical microscopy, XRD and XRF.
- **Image Processing**: including digital and optical camera. Semi-automated image analysis is available

Mechanical Properties

- **Compressive testing machines** (Structures Division)
- **Micro-hardness** measurements (Polymers Division)
- **Nano-indenter**
- **Stress measurements** of a confined specimen for ASR or sulfate attack studies

Transport Properties

- **Gas Permeability Equipment**
- **X-ray Absorption Unit (new in 2001)** for monitoring water movement in materials

Systems characterization

- **Atomic Absorption**
- **Infrared spectrometer**
- **Ion Chromatograph (new in 2001)**
- **Thermogravimetric Analysis**

- **Differential Scanning Calorimeter**
- **Differential Thermal Analysis**
- **Automated X-ray florescence spectrometer** for bulk chemical analysis (Analytical Chemistry Division)
- **Automated high-temperature furnace** to 1600 C; used to determine for instance the degree of hydration of cement paste, or to synthesize pure clinker phases or clinker
- **Automated microprobe** utilizing multiple wavelength spectrometers (Analytical Chemistry Division)
- **Dunouy Tensiometer** for measurement of the surface tension of solutions.
- **Heiden Sorption Analyzer** for measurement of absorption/desorption isotherms.

Rheology

- **Parallel plate fluid rheometer**: stress or strain controlled
- **Controlled temperature and speed mixer**: This mixer is designed according to the specifications developed by PCA/CTL and can be used for paste and/or mortar
- Other tools for flow measurement in cement paste or mortar: Marsh cone, flow cone, mini slump
- Tools to measure concrete workability: Slump and modified slump test.

Sample preparation laboratory:

- Fully equipped laboratory to prepare and cure mortar or cement paste specimens including Hobart mixers, flow table, vibrating table
- Environmental chambers: 3 cabinets (temperature and RH controlled) and one walk-in (temperature controlled)
- Water baths: 4 table top and one floor unit
- pH controlled units
- Length measurement devices

Computational Facilities (Building Materials Division):

- DEC Alpha 2100 Server (2 processors, 512 MB RAM)
- SGI Indigo 2
- Windows and LINUX-based PCs
- Linux-based cluster with 16 nodes (32 processors- **new in 2001**)

Equipment in the Dispersion & Fine Particle Labs, Ceramics Division (MSEL):

Responsible: Vince Hackley

- **Matec Applied Science ESA-8000** (electroacoustic analyzer particle mobility)
- **Colloidal Dynamics Acoustosizer** (electroacoustic spectrometer for particle size and zeta potential)
- **Dispersion Technology DT-1200** (acoustic spectrometer with CVI for particle size and zeta potential)

- **Malvern Zetasizer 3000HS** (microelectrophoresis and QELS particle sizer)
- **Rheometrics 2000** (controlled stress dynamic rheometer)
- **Brookfield DV-II+ Viscometer**
- **Coulter LS230** (laser diffraction particle sizer)
- **Micromeritics Sedigraph 5100** (gravitational sedimentation particle sizer)
- **Quantachrome AUTOSORB-1** (multi-point surface analysis system for BET surface area and porosity)
- **Quantachrome AUTOSCAN 60** (mercury porosimeter)
- **Perkin-Elmer 330 Spectrophotometer**
- **Rosemount Dohrmann DC-80 Total Organic Carbon Analyzer**
- **AMRAY 1830 Scanning Electron Microscope**
- **Beckman J2-series High Speed Centrifuge**

Information Technology Laboratory Central Hardware Facilities (ITL):

Responsible: Steve Satterfield

Computational Resources:

- IBM SP2:
  - 48 node, 80 CPU and a Scalable POWERparallel Switch.
  - Each SP2 node has at least 512 MB of memory and 2.5 GB of temporary disk space.

- Four SGI Origin 2000 machines:
  - 8 196 MHz R10000 CPUs, 8 GB of memory, 120 GB of disk space
  - 32 250 MHz R10000 CPUs, 32 GB of memory, 96 GB of disk space
  - 32 300 MHz R12000 CPUs, 32 GB of memory, 193 GB of disk space
  - 32 300 MHz R12000 CPUs, 32 GB of memory, 193 GB of disk space

- One SGI Cluster
  - five dual processor R10000 CPUs, with 4 GB of memory

- Three Linux Clusters
  - sixteen 400 MHz Pentium IIs connected by a Fast Ethernet network. Each node has 1 CPU with 256 MB of RAM and 6 GB of local disk storage.
  - forty-eight 500 MHz Pentium IIIs connected by a Fast Ethernet network. Each CPU has at least 256 MB of RAM and 6 GB of local disk storage.
  - 128 Pentium IIIs connect by a Fast Ethernet network Each CPU has 1 GB of RAM and 6 GB of local disk storage

Visualization Resources:

- One SGI Onyx with three graphics pipelines, twelve R12K 500 MHz CPUs, 12 GB of memory
- One RAVE two-wall immersive environment with Crystal Eyes software/hardware with head tracking

VCCTL TECHNICAL AND SOFTWARE NOTES

"Providing the technical basis for quantitative materials characterization and critical guidance for using the VCCTL computer programs"

VCCTL Tech Note

Technical Note VCCTL-01: Estimation of the Degree of Hydration of Portland Cement by Determination of the Non-Evaporable Water Content

Technical Note VCCTL-02: SEM/X-ray Imaging of Cement Powders

Technical Note VCCTL-03: Quantitative Determination of Calciumsulfate Dihydrate and Calciumsulfate Hemihydrate in Cement by Means of Thermogravimetric Analysis (from Dyckerhoff Zement)

Technical Note VCCTL-04: Estimation of the Degree of Hydration of Cement by Measurement of Chemical Shrinkage

VCCTL Software Note

Software Note VCCTL-01: *CONCRETEVIEW* software

Software Note VCCTL-02: Software for Hydration/Drying of 2 mm Thick Microstructures

VCCTL PUBLICITY

Concrete Technology Today- PCA (December, 2000 issue) – The Future of Materials Testing? Virtual Testing of Concrete Will Save Time and Money

NIST Update (February 20, 2001 issue) – Virtual Lab Consortium to Test Concrete and Cement Formulas

NIST Technology at a Glance (Spring 2001 issue) – Co-Op Corner, Virtual Cement Laboratory

Civil Engineering (June, 2001 issue)- Virtual Concrete Lab Saves Time, Reduces Material Cost

Engineering News Record (June 25, 2001 issue)- Website Aims to Cut Concrete Analysis (available at http://www.enr.com/itnews/it62501e.asp)

Government Computer News (August 27, 2001 issue)- NIST Team Sees in Stereo (available at http://www.gcn.com/20_25/news/16941-1.html)

The Concrete Producer (October, 2001 issue)- E-Concrete: Let's Try a Little Left Thinking

Concrete Construction (November, 2001 issue)- Virtual Concrete Laboratory

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Portland Cement Association
Dr. Paul Tennis

W.R. Grace & Co- Conn.
Dr. Vijay Gupta
Dr. David Myers

FOR MORE INFORMATION

1) Examine version 1.0 of the VCCTL software available at:
   http://vcctl.cbt.nist.gov

2) Visit the VCCTL consortium information web site at:
   http://www.bfrl.nist.gov/862/vcctl

3) Contact the VCCTL Consortium Manager at:

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