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# Virtual Testing in a Cement Plant

## A tool for producing customized products

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The Siam Cement Group (SCG) is Thailand's largest industrial conglomerate. Its cement production group, now known as SCG Cement, has been producing cement for nearly 100 years. About 50% of the ASTM C150 Type I ordinary portland cement (OPC) produced by SCG Cement is consumed by CPAC Concrete Products and Aggregate Co., Ltd., a ready mixed concrete producer within the conglomerate. Other significant consumers of SCG Cement's Type I OPC are manufacturers of precast concrete products. These producers typically purchase in bulk—cement is delivered in trucks and pneumatically conveyed into silos. SCG Cement also supplies Type I OPC in sacks to smaller concrete producers.

Each market segment has different cement performance requirements. For example, a typical precast/prestressed concrete producer in Thailand normally requires short working time, very low slump, and rapid strength gain so that the concrete can be stressed within 18 hours of placement. In contrast, a typical ready mixed concrete supplier needs a consistent and sufficiently long setting time, good workability, minimal slump loss, and a reliable 28-day target strength. Until recently, Type I OPC has been viewed as a static, uniform commodity, with cement manufacturers largely supplying a single Type I OPC to all market segments. Therefore, each customer has typically been forced to tune the cement's performance by using chemical admixtures. This approach is not entirely satisfactory because it adds considerable costs to the final product and shifts some of the burden of assuring cement performance to the customer.

Some cement manufacturers are beginning to address these issues by transitioning cement from a uniform commodity to a differentiable, customized product. The practices and needs of each particular market segment are being carefully analyzed to create a finely tuned cement product for that market. Nevertheless, to be successful, this strategy requires an agile production environment in which manufacturing parameters can be rapidly adjusted to produce materials with reliable characteristics that will translate to assured performance for a given market segment. SCG Cement strives to achieve this kind of adaptability by facilitating collaboration between its customers and SCG Cement's research arm, Siam Research and Innovation Co., Ltd. (SRI). This article describes one such collaborative effort that leveraged numerical modeling and basic materials engineering experience to predict the relationships between cement characteristics and performance and efficiently find promising combinations of parameters for production. This Integrated Computational Materials Engineering (ICME) approach has been applied to other materials[1] and is in the spirit of the U.S. Materials Genome Initiative.[2]

### Translating Needs into Optimized Parameters

For a given clinker composition, a traditional cement plant can adjust two primary parameters to optimize Type I OPC performance: sulfate balance and fineness. Calcium sulfates, whether in the form of gypsum (dihydrate), bassanite (hemihydrate), or anhydrite, are used to regulate the reactivity of calcium aluminates in the clinker, thereby preventing false set or flash set. The total amount of sulfates, as well as the blend of the different sulfate forms, therefore significantly impacts both early-age properties, such as setting time and workability, and longer-term properties, such as 28-day compressive strength. The ability to control the amount of the different forms of sulfates depends on the available raw materials and control of grinding mill parameters such as temperature, residence time, and circulation factor.

Cement fineness influences performance at least as much as sulfate content. Finer cements require more grinding (lowering throughput and increasing energy costs), but if the cement is not sufficiently fine, the resulting product will not hydrate properly. Fineness is often reported as a single number, such as Blaine fineness (ASTM C204, "Standard Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus"), but the characteristics of the powder are more complicated and important than a single number can capture. Powders with broader particle size distributions (PSDs), typically measured by laser diffraction, tend to demonstrate better packing and result in decreased water demand. Lower water demand is preferable for optimizing cement performance, so there is a delicate balance between having a sufficient fraction of fine particles for good particle packing and having such a high fraction that water demand becomes excessive.[3]

To illustrate this point, PSDs of two cements with very similar Blaine fineness (330 and 335 $m^2/kg$) are compared in Fig. 1. The powder from Mill A is somewhat finer and has a broader PSD than the powder from Mill B. The greater fraction of particles in the size range of 2 to 10 µm makes the cement from Mill A have a water demand that is 10 $L/m^3$ (2 $gal./yd^3$) higher than the cement from Mill B—an increase sufficient to result in increased segregation, reduced concrete consistency, and diminished overall performance.

Unfortunately, it is quite challenging to optimize performance at the plant scale because the viable combinations of sulfate content and PSD are endless. While lab mills can be used for preliminary investigations, optimization results obtained from lab mills often are not

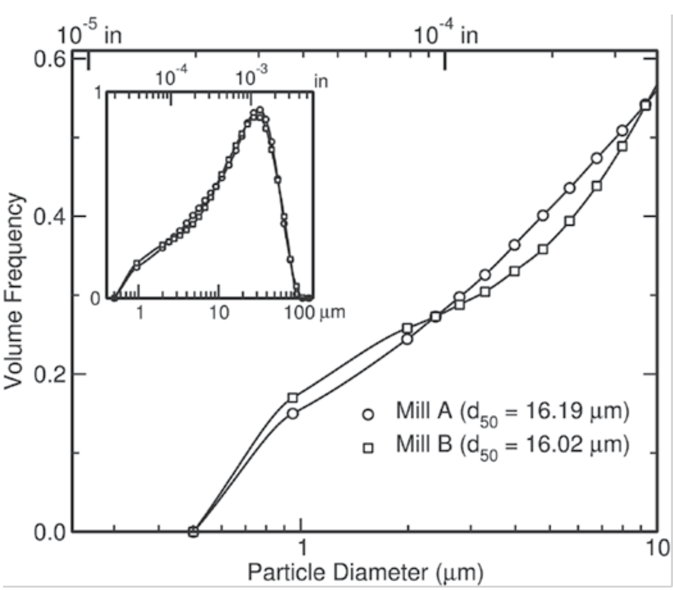


**Fig. 1: PSD plotted as a volume frequency or probability distribution of Type I OPC produced from two different mills, each producing the same nominal Blaine fineness. The greater fraction of particles in the size range of 2 to 10 µm makes the cement from Mill A have a water demand that is 10 $L/m^3$ (2 $gal./yd^3$) higher than the cement from Mill B. The inset shows the full PSD. Variation in the PSD over six repeated measurements is less than the size of the symbol in the plots**

easily correlated with results from pilot mills and production mills because lab mills cannot mimic all the variables and parameters that affect mill behavior at the plant scale. Extensive testing at the plant scale is cumbersome and cost-prohibitive, however, because of the potential loss of production and the difficulty of isolating and controlling individual test parameters. For example, different materials have different grindabilities, so changing the sulfate content of cement gives it different grinding characteristics and ultimately influences the PSD.

Table 1 shows the mineralogical composition, as measured by quantitative X-ray diffraction (QXRD), and fineness of cements that were sampled directly from the production line during a plant trial. These cements were used to prepare mortar cubes for compressive strength tests (ASTM C109/C109M, "Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)") listed in Table 2. Because of the uncontrolled variables in each step, the impact of each parameter on the strength is difficult to understand. Even when optimized values are obtained, they must be in a range where inherent production fluctuations can be tolerated without significantly impacting the optimal performance of the cement, and they must also meet industry standards. Clearly, unguided trial-and-error optimization is both challenging and costly. In light of these difficulties, our researchers have moved to the use of numerical modeling to provide guidance in the optimization process and to discover the underlying mechanisms that govern the results.

## The Role of Numerical Models

Using the considerable body of knowledge on the materials science of cementitious materials, the National Institute of Standards and Technology (NIST) has developed a range of microstructure-based models of the hydration, mechanical, and transport properties of cement, mortar, and concrete materials. A NIST Electronic Monograph (**http://concrete.nist.gov/monograph**) contains a repository of publications that document these models and the breadth of problems they can address. Beginning in 2001, many of these models were collected into an integrated software package called the Virtual Cement and Concrete Testing Laboratory (VCCTL). In the interim, the user interface for the package has advanced to the point that experienced engineers can use it with a small amount of training.

VCCTL comprises numerical models that are rooted in basic principles of physics and chemistry. The core of VCCTL is its hydration model, which simulates the development of cement paste microstructure as the binder matures. The microstructure is modeled as a 3-D collection of small cubic volume elements, or voxels, each with an edge length of 1 µm. Each voxel is assigned a material component—for example, alite, porosity, or aggregate—and the hydration model sequentially changes the voxel component as a result of chemical reactions, such as dissolution, nucleation, and growth.

Different probabilities are assigned based on the various reactions that can happen at a given voxel based on the identity of the voxel and its neighbors. For example, bassanite dissolves more readily in water than anhydrite, so a bassanite voxel is assigned a greater probability of dissolution at each time step than a voxel of anhydrite. The voxel-based approach also naturally accommodates the influence of microstructure parameters such as PSD. For example, voxels at the surface of smaller particles are in contact with more voxels of water-filled porosity on average than those at the surface of larger particles, so smaller particles dissolve more rapidly.

The virtual testing approach embodied by VCCTL has at least two important advantages that make it attractive for supplementing tests in a cement plant. First, it provides results quite rapidly. It takes only about 6 hours to compute the 28-day elastic moduli and compressive strength of a mortar, using a material volume of 0.01 $mm^3$ on a standard desktop computer. Second, it provides immediate access to the often-complicated relationships between basic material parameters (such as water-solids ratio, PSD, sulfate type and amount, and clinker phase fractions) and a wide range of engineering properties (such as setting time, heat of hydration, elastic moduli, conductivity, and compressive strength). By providing guidance to the engineer about the sensitivity of performance characteristics to material parameters, VCCTL therefore reduces the risk of making costly mistakes in the plant.

VCCTL has been used extensively to help make sense of a wide range of experimental observations on cement, mortar, and concrete; many examples are provided in the electronic monograph (**http://concrete.nist.gov/monograph**). In this article, we focus on SRI's use of VCCTL to guide

## Table 1:
### Fineness and mineralogical composition

| Mineral compound | Mineralogical composition expressed as % of total mass | | | | | | |
|---|---|---|---|---|---|---|---|
| | Blaine fineness, m²/kg | | | | | | |
| | 330 | 347 | 356 | 327 | 338 | 334 | 347 |
| Alite | 68.2 | 64.5 | 63.1 | 67.1 | 64.7 | 64.2 | 65.3 |
| Belite | 4.9 | 8.0 | 6.9 | 4.2 | 6.5 | 6.3 | 5.7 |
| Aluminate | 7.6 | 7.8 | 7.6 | 8.7 | 8.6 | 8.3 | 7.9 |
| Ferrite | 9.8 | 9.4 | 9.9 | 9.6 | 8.8 | 9.5 | 8.7 |
| Gypsum | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.6 | 0.0 |
| Bassanite | 3.5 | 4.3 | 5.4 | 4.2 | 4.4 | 4.0 | 3.9 |
| Calcite | 3.8 | 3.9 | 5.0 | 4.2 | 4.4 | 4.0 | 3.9 |
| Free lime | 0.4 | 0.2 | 0.3 | 0.3 | 0.5 | 0.2 | 0.3 |
| Portlandite | 0.7 | 1.0 | 0.6 | 0.7 | 1.0 | 1.1 | 1.4 |
| Periclase | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Quartz | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 |
| Arcanite | 0.7 | 0.7 | 0.9 | 0.8 | 0.9 | 0.8 | 1.0 |
| Aphthitalite | 0.0 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |

Note: Expressed as % of total mass, determined by QXRD of cement powder from the plant trials. Mean values are reported with typical standard deviations of less than 2% of the mean for fineness and 4% absolute for composition data. Round-off error may cause the sum of the composition to vary between 99.8 and 100.1%

## Table 2:
### Mortar strength values for cement samples analyzed in Table 1

| Sample | Blaine fineness, m²/kg | Bassanite content, % | Measured mortar strength at indicated age, MPa (ksi) | | | |
|---|---|---|---|---|---|---|
| | | | 1 day | 3 days | 7 days | 28 days |
| 1 | 330 | 3.5 | 13.7 (1.99) | 25.1 (3.64) | 33.9 (4.92) | 42.0 (6.09) |
| 2 | **347** | **4.3** | **14.5 (2.10)** | **27.2 (3.94)** | 35.1 (5.09) | 43.5 (6.31) |
| 3 | **356** | **5.4** | 13.8 (2.00) | 26.3 (3.81) | **35.6 (5.16)** | **44.4 (6.44)** |
| 4 | 327 | 4.2 | 13.3 (1.93) | 24.6 (3.57) | 32.7 (4.74) | 41.9 (6.08) |
| 5 | 338 | 4.4 | 13.6 (1.97) | 24.4 (3.54) | 35.5 (5.15) | 42.9 (6.22) |
| 6 | 334 | 4.0 | 11.3 (1.64) | 23.2 (3.36) | 31.8 (4.61) | 39.4 (5.71) |
| 7 | 347 | 3.9 | 13.6 (1.97) | 24.6 (3.57) | 34.0 (4.93) | 43.0 (6.24) |

Note: Boldface values correspond to the maximum strength in each column. Simulation data have an uncertainty of less than 5% of the mean strength calculated from three independent simulations parameter optimization for sulfate balance and cement fineness to customize SCG Cement's products.

## Requirements for VCCTL Modeling

The models used in VCCTL operate on and derive their predictive power from the details of cement paste microstructure and how the microstructure changes with continued hydration. Therefore, accurate knowledge of many aspects of the starting materials—both cement and aggregate—is crucial to the models' predictive capabilities. Prior research[4] has revealed the importance of knowing the value of the following cement powder variables, as a bare minimum, for input into the model:

- Volume fraction of each component in the material, including mineral admixtures and each of the various calcium sulfate carriers (for example, gypsum, bassanite, and anhydrite);
- Surface area fractions of each component of the clinker, as a measure of how phases are distributed within cement particles; and
- The cement PSD, which is the primary structural variable controlling the reactivity.

Within this minimal set of cement variables, the volume fraction (or, alternatively, the mass fraction) of each crystalline phase can be obtained by QXRD of the powder using Rietveld refinement, which is by now a fairly common characterization method at cement plants. Surface area fractions of the clinker phases, in contrast, can only be measured by scanning electron microscopy of polished sections coupled with X-ray imaging to distinguish the various phases. With a series of electron micrographs that have been segmented to identify the phases, stereological methods can be applied to determine the surface area fractions. The same micrographs can be used together with principles of stereology to calculate the volume fractions of the various cement phases, thereby providing a check on the bulk powder XRD analysis. The details of the techniques involved in this microscopic characterization of cement powders, including procedures for specimen preparation and analysis, have been comprehensively described in several publications.[4-6]

Other variables, of secondary importance for determining hydration behavior and microstructure development, can also be measured and input into the VCCTL models. Among these are the mass percentage of total alkali content that is water-soluble (ASTM C114, "Standard Test Methods for Chemical Analysis of Hydraulic Cement") and particle shape.[7] But even within the minimal set of required input, several of the material parameters may not be routinely measured in cement plants, such as the full PSD of the powder, the type and quantity of each sulfate carrier, and both the volume fraction and surface area fraction of the major clinker phases. Nevertheless, SRI's experience is that the return on investment is easily great enough to justify the effort needed to make these kinds of measurements.

When working with virtual mortars or concrete instead of cement paste, certain properties of the coarse and fine aggregate sources are required for input to VCCTL, including the aggregate grading and the bulk and shear moduli for each aggregate component. The software interface provides users with the capability to create new virtual cement materials by inputting or uploading the cement material parameters and aggregate properties that are required for accurate simulations.

## Results from Modeling

In an initial effort to verify the utility of VCCTL in the SCG Cement plants, SRI researchers used the software to help optimize sulfate content and cement fineness for a clinker of fixed composition. Plant engineers and researchers worked together to identify ranges of the sulfate and fineness parameters pertinent to the plant trials. Input parameters were not estimated but taken directly from analysis of actual plant samples. The plant engineers established an acceptable range of sulfate content of 2.0 to 4.0% by mass of cement. This range was discretized into five values in increments of 0.5%. Due to the nature of the grinding process in the plant, most of the sulfates are in the form of bassanite (Table 1). The cement is typically ground to a Blaine fineness of 325 to 375 $m^2/kg$. The fineness values and the corresponding PSDs were chosen to mimic actual samples collected during plant trials and the range was discretized to be as follows: 325, 335, 345, 353, and 373 $m^2/kg$. Although results were reported in terms of fineness, the PSD of each powder was measured for input to VCCTL. A total of 25 combinations of fineness and sulfate level were modeled.

The SCG plant maintains a standard practice of measuring product quality in terms of mortar strengths at 3, 7, and 28 days. Table 3 shows VCCTL predictions of mortar strengths at those ages, normalized to the strength at 2.0% (by mass) sulfate content and 325 $m^2/kg$ fineness. The use of normalized strength is a common practice in our production process because actual strength values would heavily depend on the clinker characteristics, which vary from time to time. In the production line, however, the greatest interest is for strength improvement and normalized values show that most clearly. No single optimal sulfate content is observed for maximizing 3-day strength, but optimal sulfate contents are clearly observed for the strengths at 7 and 28 days. Furthermore, increasing the cement fineness showed diminishing returns, with negligible strength gains once the fineness exceeds 353 $m^2/kg$. Therefore, it is not beneficial to produce cements of higher fineness, especially because finer cements also have higher water demands.

The question remains as to what primary mechanism or mechanisms control the observed behavior. VCCTL results indicated a progressively greater degree of hydration with increasing cement fineness and with lower sulfate content, even though there is no correlated strength gain. Therefore, degree of hydration alone cannot account for the optimal sulfate phenomenon in these cements. Similarly, C-S-H content, the glue that is responsible for strength gain in the binder, is predicted to increase continually with higher fineness and lower sulfate content. Therefore, optimal sulfate is not associated with a maximum in C-S-H volume fraction.

Nevertheless, the total capillary porosity did reach a minimum value that coincided with the optimal sulfate value. Insufficient sulfate dosages tend to decrease the quantities of ettringite and monosulfate in the hydrated microstructure in favor of AFm phases that have a lower molar volume and plate-like morphology that cannot efficiently fill space. Furthermore, hydration of silicate minerals in the clinker is retarded when sulfate levels are too low.[8] Both of these effects produce greater capillary porosity at any time when sulfate levels are below the optimum. When sulfate levels are too high, however, the excess sulfate cannot promote the formation of additional solid hydration products. Instead, the continued dissolution of the extra gypsum or bassanite particles leaves behind void space that is not completely filled by hydration products. Again, the result is a greater capillary pore volume than that produced at the optimum sulfate level. The minimum in capillary porosity at the optimum sulfate dosage correlates directly with a maximum in compressive strength.[9,10]

The foregoing results refer to VCCTL predictions for the cement that was used in the trials. Nevertheless, if VCCTL is to be used as a tool in optimization of cement parameters, its predictions should be borne out in actual plant trials. Table 3 shows that the optimum $SO_3$ content at 7 days is 3.0 and at 28 days is 3.5 at the higher fineness values used in actual production. In fact, the agreement is remarkably good, considering production line requirements: plant trials indicated that the optimum combination of $SO_3$ content and cement fineness is around 2.6% sulfate content along with a fineness of 360 $m^2/kg$.

The lower production set point for $SO_3$, compared to the VCCTL calculated optimum values, is partly due to the need to stay below the ASTM C150/C150M, "Standard Specification for Portland Cement," upper limit of 3.5% for clinkers with $C_3A$ contents exceeding 8%. The lower value allows for some heterogeneity in the $SO_3$ distribution while still ensuring that the upper limit is not exceeded. In addition, the lower production value helps guard against the risk that variations in clinker chemistry during normal production could result in an over-sulfated system. This result from the plant trials demonstrates that VCCTL can accurately predict cement characteristics for optimum performance and that such characteristics are both technically and economically viable for large-scale production. In addition, the models not only predict but also explain the observed behavior—information that can be valuable during future changes in production or optimization efforts.

## Long-term Planning

Changes in customer requirements in different market sectors, as well as the environmental demands on our industry, are leading to the development of products that are pushing normal plant operations into uncharted territories. To accommodate these demands with long-term sustainable solutions, a wide range of ideas must be explored, which may not be possible to test experimentally.

Virtual testing will allow engineers to explore risky but possibly very rewarding solutions that will improve short-term plant operations and long-term planning. More complex systems than OPC, such as portland limestone cement or blended cements, will gain even greater benefits from numerical modeling, because such systems inherently have more production parameters that must be optimized (for example, the PSD of each component). Sulfate optimization at the cement plant may be of limited value at ready mixed concrete batching plants when supplementary cementitious materials (SCMs) are added directly to the mixture. VCCTL can potentially provide guidance as to whether a certain fly ash is a suitable match for a particular cement, although such an application would require a more detailed characterization of the fly ash than is performed in practice.

For long-term planning in cement production, the challenge is the effective use of innovative alternative fuels and lower-quality raw materials, which will result in the production of clinkers with a lower lime saturation factor. VCCTL can be a very useful tool in finding acceptable ranges of cement production parameters, given a substantially different clinker characteristic, such that the resulting product continues to meet customer needs and industry specifications. Although these parameters still need to be finalized in plant trials, the knowledge of how and why each parameter impacts the cement performance will help production engineers navigate through the optimization process. Our researchers have adopted the use of numerical modeling to map out these uncharted territories, enabling SCG Cement to develop greater versatility while minimizing risk for customers.

### Table 3:
### VCCTL predictions of compressive strength at 3, 7, and 28 days, using ASTM C109

| Sulfate content, % | Compressive strength at 3 days expressed as % of strength standard | | | | |
|---|---|---|---|---|---|
| | Blaine fineness, m²/kg | | | | |
| | **325** | **335** | **345** | **353** | **373** |
| **4.0** | 92.9 | 92.2 | 93.8 | 101 | 99.8 |
| **3.5** | 98.1 | 101 | 102 | 106 | 105 |
| **3.0** | 99.7 | **102** | 88.6 | **109** | **109** |
| **2.5** | **102** | 101 | 99.6 | 108 | 101 |
| **2.0** | 100 | 102 | **103** | 108 | 105 |
| **Sulfate content, %** | **Compressive strength at 7 days expressed as % of strength standard** | | | | |
| | **Blaine fineness, m²/kg** | | | | |
| | **325** | **335** | **345** | **353** | **373** |
| **4.0** | 92 | 92 | 93.4 | 101 | 101 |
| **3.5** | **103** | **103** | **105** | 107 | 106 |
| **3.0** | 102 | **103** | 92 | **108** | **108** |
| **2.5** | 102 | 102 | 100 | 106 | 100 |
| **2.0** | 100 | 102 | 103 | 106 | 104 |
| **Sulfate content, %** | **Compressive strength at 28 days expressed as % of strength standard** | | | | |
| | **Blaine fineness, m²/kg** | | | | |
| | **325** | **335** | **345** | **353** | **373** |
| **4.0** | 104 | 102 | 104 | 107 | 107 |
| **3.5** | **108** | **108** | **110** | **111** | **110** |
| **3.0** | **108** | **108** | 97.2 | 110 | **110** |
| **2.5** | 103 | 102 | 101 | 106 | 99 |
| **2.0** | 100 | 101 | 102 | 104 | 103 |

Note: At 3, 7, and 28 days, using ASTM C109, normalized to the strength calculated for sulfate content of 2.0% and fineness of 325 m²/kg (strength standard). Boldface values correspond to the maximum relative strength in each column. Simulation data have an uncertainty of less than 5% of the mean strength calculated from three independent simulations.

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Note: Additional information on the ASTM standards discussed in this article can be found at www.astm.org.

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