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Keynote Paper

ADVANCES IN CONCRETE MIXTURE OPTIMISATION

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ABSTRACT. The complexity of high performance concrete (HPC) mixture designs continues to increase, along with the number of criteria that a particular mixture must satisfy. Robust statistical methods exist that can be used to not only determine the mixture for a concrete that meets specifications, but does so while satisfying a number of additional user-specified constraints. Two such techniques are the mixture method and the response surface method of experimental design. These techniques are discussed and compared. Future developments in concrete mixture optimization based upon materials science-based models are discussed.

Keywords: High performance concrete (HPC), Materials science, Mixture design, Mixture optimization, Response surface methodology (RSM)

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INTRODUCTION

As higher and higher performance is sought from concrete, obtaining the proper mixture proportion to achieve specific objectives is becoming more difficult and useful tools are needed to aid the process. The Partnership for High Performance Concrete Technology (PHPCT) of the National Institute of Standards and Technology (NIST) and the Federal Highway Administration (FHWA) seek to facilitate the use of high performance concrete (HPC) in both public and private construction, and are currently developing tools for optimizing HPC mixture proportions to meet a number of performance criteria (user-specified constraints) simultaneously. These performance criteria could include fresh concrete properties such as viscosity, yield stress, setting time, and temperature; mechanical properties such as strength, modulus of elasticity, creep and shrinkage; and durability-related properties such as resistance to freezing and thawing, abrasion, or chloride penetration.

The American Concrete Institute (ACI) defines HPC as "concrete meeting special combinations of performance and uniformity requirements that cannot be achieved routinely using conventional constituents and normal mixing, placing, and curing practices." [1]. Since fewer, or possibly no, prescriptive constraints, such as minimum cement contents or maximum water-cement ratios, will be included in performance specifications, a concrete producer or materials engineer will have greater than usual latitude in selecting constituent materials and defining their proportions. At the same time, the task of achieving the design specifications has become more complex. HPC mixtures are usually more expensive than conventional concrete mixtures, since they usually contain one or more of the following: (1) more cement, (2) higher dosages of chemical admixtures, and (3) mineral admixtures. The desire to optimize concrete mixture proportions, by meeting the performance criteria at the lowest cost, increases as the cost of materials increases. Furthermore, as the number of constituent materials increases, the problem of identifying optimal mixtures becomes increasingly complex. Not only are there more materials to consider, but more potential interactions among materials. Combined with several performance criteria to meet, the number of trial batches required to find optimal proportions using traditional methods could become prohibitive. Statistical and computational tools are required to provide cost-effective means of formulating optimized concrete mixtures.

The process of concrete mixture proportioning typically involves the following steps:

1. Identifying a starting set of mixture proportions

2. Performing a series of trial batches, starting with the mixture identified in Step 1, and adjusting the proportions in subsequent trial batches until all criteria are satisfied. Typically, this is performed by changing one component at a time.

The remainder of this paper discusses two types of tools that could be used to improve the process of mixture proportioning. The first of these is the application of statistical methods for product optimization. The second, a not-yet-fully-realized goal of the NIST program, is the application of materials science-based models [2] to predict the performance of concrete mixtures from knowledge of the properties and proportions of the ingredients and the processing of the mixtures [3].

APPLICATION OF STATISTICAL METHODS TO CONCRETE MIXTURE PROPORTIONING

Current practice in the United States for developing new concrete mixtures usually relies upon historical information (i.e., what has worked for the producer in the past) or the guidelines for mixture proportioning outlined in ACI 211.1 [4]. While both methods can yield a starting point for trial batches, neither method is a comprehensive procedure for optimizing mixtures. Not only does ACI 211 not account for interacting effects among the concrete constituents, there is no means by which one can efficiently achieve an optimized mixture for a given criterion. In contrast, statistical experimental design methods are rigorous techniques for both achieving desired properties and determining an optimized mixture for a given constraint, while minimizing the number of trials. They are used widely in industry to optimize products and processes [5].

Following the ACI 211 guidelines, an engineer would select and run a first trial batch (using proportions selected using ACI 211 or historical data), evaluate the results, adjust the proportions of various components and run further trial batches until all specified criteria were met. Employing statistical methods in the trial batch process would not change the overall approach, but would change how trial batching is done and speed the process. Rather than selecting one starting point, a set of trial batches covering a chosen range of proportions for each component is set up according to established statistical procedures [5]. Trial batches are performed and results are analyzed using standard statistical methods that yield reliable estimates of parameters from empirical models for each performance criterion. Each response, such as strength, slump, or cost, is expressed as an algebraic function of factors such as water-cement ratio (w/c), cement content, chemical admixture dosage, pozzolanic replacement, etc. Once a response can be characterized by an equation, any number of analyses are possible. For instance, the user could determine which mixture proportions would yield a desired response. Similarly, the user could optimize any response function subject to constraints on the others. For example, one could determine the lowest cost mixture with strength greater than the specified strength. A method for optimizing several responses simultaneously is described later in the paper.

Efforts have also been made to develop mixture proportioning methods based on mechanistic (or semi-mechanistic) models [6]. In this approach, models are developed from results of fundamental and applied materials research, and the user does not perform a series of trial batches from which empirical models are estimated. This approach has the advantage of eliminating the need for trial batches to obtain the models; however, some trial batches would most likely be needed to adjust proportions because of differences in a user's specific materials. It is unlikely that a mechanistic model would be able to account for all possible differences among materials from various localities. The advantage of the trial batch approach is that the project-specific materials are used and accounted for in the model.

The statistical approach has an additional advantage that is often overlooked in mixture design procedures: the expected responses can be characterized by an uncertainty. This has important implications for both mixture specification and for production. One could use the empirical model equations to determine a mixture design that yielded a desired strength. However, the model equation would only give the expected mean strength; if replicate mixtures were made, the model equation would predict the *mean* value. For producers to be relatively sure that most of the on-site tests would comply with the specifications, they would select target values for the mean strength to account for the variability and to ensure that, say, 95 percent of the time the concrete performance would be in compliance.

Background on Statistical Approaches to Optimization

There exist two primary approaches to the general problem of optimizing a mixture whose properties depend on the proportions of the component materials: the *mixture approach* and the *response surface approach* [5]. Each technique has advantages and disadvantages.

Mixture approach

Using a mixture approach, the total amount (mass or volume) of the product is fixed, and the settings of each of the q components are proportions. Because the total amount is constrained to sum to one, only q-1 of the component variables are independent.

As a simple example of a mixture experiment, consider concrete as a mixture of three components: water (x_1), cement (x_2), and aggregate (x_3), where each x_i represents the volume fraction of a component. Assume the coarse-to-fine aggregate ratio is held constant. The volume fractions of these components sum to one,

$$x_1 + x_2 + x_3 = 1 \tag{1}$$

and the region defined by this constraint is the triangle, or three-component simplex, shown in Figure 1a. The axis for each component x_i extends from the vertex it labels ($x_i = 1$) to the midpoint of the opposite side of the triangle ($x_i = 0$). The vertex represents the pure component. For example, the vertex labeled x_1 is the pure water mixture with $x_1 = 1$, $x_2 = 0$, and $x_3 = 0$, or (1,0,0). The coordinate where the three axes intersect is (1/3,1/3,1/3) and is called the *centroid.* A good experiment design for studying properties over the entire region of a three-component mixture would be the simplex-centroid design shown in Figure 1b (this example is included as an illustration only, since much of this region would not represent either feasible or workable concrete mixtures). The points shown in Figure 1b represent mixtures included in the experiment and include all vertices, midpoints of edges, and the overall centroid. For each mixture, all properties of interest would be measured, and empirical models for each property as a function of the components would be determined from regression analysis.

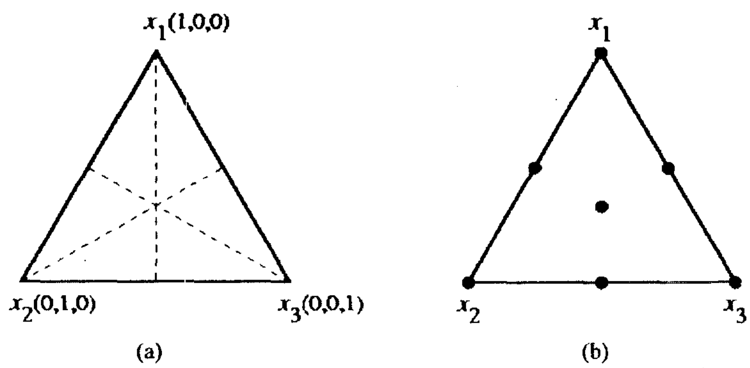

Figure 1 Three-component mixture plans: a) experimental region, and b) layout of simplex-centroid experiment design

Since feasible concrete mixtures do not exist over the entire region shown in Figures 1a and 1b, a subregion of the full simplex that contains the range of feasible mixtures must be defined by constraining the component proportions. An example of a possible subregion for the three-component example is shown in Figure 2. It is defined by the following volume fraction constraints (x_1 = water, x_2 = cement, x_3 = aggregate):

$$0.15 \leq x_1 \leq 0.25$$
$$0.10 \leq x_2 \leq 0.20$$
$$0.60 \leq x_3 \leq 0.70$$

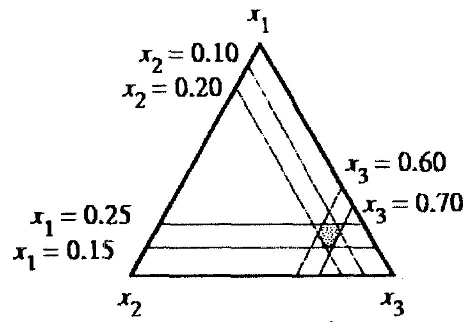

Figure 2 Example of constrained experimental region for a mixture with three components

In this case simplex designs are no longer appropriate and other designs are used [5].

The advantage of the mixture approach is that the experimental region of interest is more naturally defined; however, analysis of the results is more complicated, especially if the number of components is greater than three, as it usually will be.

Response surface approach

In the response surface approach, the q components of a mixture are reduced to $q-1$ independent variables by using the ratio of two components as an independent variable. In the case of concrete, the w/c ratio is a natural choice for this ratio variable. As a simple example, consider a concrete mixture composed of four components: water, cement, fine and coarse aggregate. Three factors, or independent variables, x_k, that can be selected to describe this system are x_1 = w/c ratio (by mass), x_2 = fine aggregate volume fraction, and x_3 = coarse aggregate volume fraction. Reasonable ranges for each variable might be:

$$0.40 \leq x_1 \leq 0.50$$
$$0.25 \leq x_2 \leq 0.30$$
$$0.40 \leq x_3 \leq 0.45$$

To simplify calculations and analysis, the actual variable ranges are usually transformed to dimensionless coded variables with a range of ± 1. In this example, the actual range of $0.40 \le x_1 \le 0.50$ would translate to a coded range of $-1 \le x_1 \le 1$. Intermediate values of x_1 would translate similarly (e.g., the actual value of 0.45 would translate to a coded value of zero).

Suppose also that the specifications for this mixture require a slump of 75 mm to 150 mm and a 28-day strength of at least 30 MPa. These specified properties are the responses, or dependent variables, y_i, which are the performance criteria for optimizing the mixture.

A common response surface experimental plan that could be used in this scenario is a *central composite design* (CCD), illustrated schematically in Figure 3. The CCD for $k = 3$ independent variables consists of eight (2^k) factorial points (filled circles in Figure 3) representing all combinations of coded values $x_k = \pm 1$, six ($2k$) axial points (hollow circles in Figure 3) at a distance $\pm\alpha$ from the origin, and at least 3 center points (hatched circle in Figure 3) with coded values of zero for each x_k. The value of α is usually chosen to make the design rotatable (implies that at locations equidistant from the origin, predicted values should have equal variance) but there could be valid reasons for selecting other values for α [5].

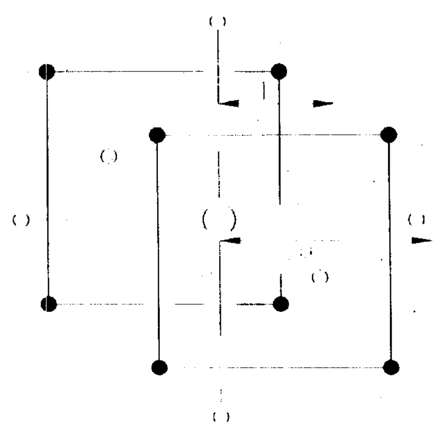

Figure 3 Central composite design for three independent variables

Empirical models

Use of an appropriate mixture experiment design or a CCD allows estimation of a full quadratic model for each response. Equation 2 shows a full quadratic model for $k = 3$ independent variables:

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + e \quad (2)$$

In Equation 2, the ten coefficients are represented by the b_k, and e is a random error term representing the combined effects of variables not included in the model. The interaction terms $x_i x_j$ and the quadratic terms x_i^2 in Equation 2 account for curvature in the response surface, which is often present when a response is at or near a maximum or minimum in the region of interest. A model with only linear terms would be sufficient if curvature was not present, and the factorial portion of the CCD is a valid design by itself in that case. Often, the presence or absence of significant curvature is not known with certainty at the start. An advantage of the CCD over the mixture approach is that the CCD can be run sequentially in two blocks. The first block would consist of the factorial points (all combinations of $x_i = \pm 1$) and some center points (at the origin), and the second block would consist of the axial points (points along each axis at distance α from the origin) and additional center points. This approach allows analysis of the factorial portion before the axial portion is run, providing an indication of whether the axial portion is necessary.

The number of coefficients in the quadratic model increases with k, and the number of trial batches required using a CCD begins to increase significantly for $k > 5$. Therefore, the use of a CCD to optimize a concrete mixture with six or more components may be uneconomical. In such cases, one could identify the most important factors and limit them to five or fewer. For example, if the cementitious materials and chemical admixtures were the most important components, they would be varied, while the amounts of coarse and fine aggregate would be held constant.

Application of the Response Surface Approach to Concrete Mixtures

As part of a current research project, NIST and FHWA are developing an interactive website that can be used to optimize concrete mixture proportions using the response surface approach. As part of this project, laboratory experiments were conducted using both the mixture approach and the response surface approach. Although both give comparable results, it was concluded that the response surface method is easier to use, and the interpretation is more straightforward. The following section describes the major steps in a response surface approach to mixture proportioning. These steps include planning (experiment design), executing trial batches, fitting and validating models, and determining optimal mixture proportions.

Experiment design

The first step in the planning process is to define the performance criteria to be met. These are usually defined in project specifications and might include (for example) slump range, fresh air content range, and minimum strength. Once the criteria are established, the next step is to select the materials to be used. Knowledge and experience are necessary here. If possible, the producer will want to use materials that he normally uses and stocks, but it is necessary to be confident that the performance criteria can be met using those materials. Otherwise, other locally available materials, or possibly special materials, will have to be procured. Comparisons of different materials (e.g., several possible choices of cement) would not usually be included in the response surface approach; therefore, if such a comparison is needed, it must be done ahead of time.

Once materials are selected, the independent variables must be chosen. One of these variables will be w/c ratio, and the rest will be volume or mass fractions of materials whose proportions will be varied. Which materials to vary will depend on the overall goal of the project and the budget allocated for mixture proportioning. Since the number of trial batches required increases exponentially ($2^k + 2k$) with k, making trial batches usually becomes prohibitive if more than six components are varied. In this case the most important components (those thought to influence properties significantly) should be identified.

Once materials are selected, the ranges for their proportions must be determined. ACI 211 or other guidelines and historical mixture information, combined with experience and knowledge, can be used to identify a starting point. The selection of ranges is important: too narrow a range may result in inability to meet all performance criteria simultaneously, too wide a range may fail to identify the best mixture.

The ranges can be defined in terms of volumes or volume fractions, mass or mass fractions. The volume fractions of all components will be constrained to sum to 1, so at some point calculation of corresponding volume fractions will be necessary. For batching, masses of materials will have to be calculated.

When the above steps are completed, the trial batching plan is constructed according to established procedures for constructing central composite experiment [5,7]. Several commercially available statistical software packages are available for this purpose, or it can be done by hand.

Execution of trial batches

Once the trial batch plan is constructed, the trial batches can be executed. As mentioned above, the CCD is usually set up in two blocks. The order of the batches within each block should be randomized. Furthermore, all batching, mixing, fabrication, and testing should be in accordance with established specifications and methods, and the same personnel should be used for the same tasks throughout the trial batching. The purpose of such steps is to minimize the effects of extraneous variables on the results. Any anomalies or aberrations in procedure or in test results should be noted.

Analysis of results

The analysis of trial batch data from tests on fresh and hardened concrete uses established graphical and numerical techniques. A graphical overview of data using scatterplots and plots of raw data helps to identify general trends and effects as well as possible outlier points (resulting from recording errors or equipment malfunction, for example).

The next step is model fitting and validation. Standard analysis of variance (ANOVA) and linear regression techniques [5] are used to estimate model parameters. A full quadratic model will usually be fit first, and t-tests on the coefficients will indicate insignificant terms which can be eliminated from the model. If the trial batches are run in sequential blocks (see above), a preliminary analysis to assess the adequacy of linear models can be performed after the batches (and required tests) in the first block are completed. As stated previously, if linear models are sufficient, the second block of trial batches may not be necessary.

Once a model is chosen, the adequacy of the model is assessed by computing residuals and examining residual plots. The residuals are the deviations of the observed data from the fitted values, and they are estimates of the error terms, e_i, in the model (see Equation 2). The error terms are assumed to be random and normally distributed, and if this assumption holds, the residuals should exhibit similar properties. Plots of the residuals against batch order (run sequence), predicted values, and other parameters should be random and without structure.

Additional quantitative checks on the adequacy of the model can be made by calculating statistical measures such as the residual standard deviation and the predicted error sum of squares (PRESS). The residual standard deviation should be close to the replicate standard deviation calculated from the center points. The PRESS statistic is a measure of how well the model fits each point in the design, with a small PRESS statistic indicating a good fit.

An additional analysis that may be valuable at this stage is to examine contour plots of the response as a function of any two variables. The contour plots are similar to topographical maps and allow the user to see how the response varies over the ranges of the chosen variables. The contour plots allow visual identification of the best settings to achieve a particular response.

Optimization

The usual goal of mixture proportioning is to identify proportions that yield concretes meeting several performance criteria (that is, constraints on several responses) simultaneously, while minimizing cost. Cost is also a response since it can be calculated as a function of the independent variables (mixture proportions). In other cases, the goal may be to maximize or minimize a particular property (say, strength) irrespective of cost. Once suitable empirical models are chosen for each response, any response can be optimized with or without constraints on the other responses. Both graphical or numerical techniques can be used for this task, however graphical methods are best used when the number of responses (including cost) is three or less. The graphical methods involve overlaying contour plots of responses with the constraints indicated, creating a feasible region that meets all of the constraints. If desired, the feasible region can be superimposed on a cost contour plot to identify the point of minimum cost.

Numerical optimization is more widely applicable. A common approach to numerical optimization uses "desirability functions," d_i, which are defined for each response (including cost) [8]. These functions vary between zero and one and can be defined in several ways. Responses that are specified by minimum or maximum values, such as strength, have desirability functions that are step functions. For strength, $d_i = 1$ above the minimum value and $d_i = 0$ otherwise. Other responses, such as slump and fresh air content, are specified by ranges. Desirability functions for these responses could be two-sided step functions if all values were equally acceptable. Alternatively, if a target value is considered most desirable, the desirability function could vary from zero at the endpoints of the range to 1 at the target value (for example, a target slump of 100 mm within an acceptable range of 75 mm to 125 mm).

The optimal set of mixture proportions is the one that maximizes the geometric mean of all of the desirability functions. Numerical search techniques are used to identify this set. After the optimal mixture is determined, the predicted values of each response are calculated and checked to see that they meet the constraints and account for the uncertainty in the model. If necessary the constraints are modified to account for the uncertainty and a revised set of optimal proportions is identified using the procedure described above.

MECHANISTIC MODELS FOR APPLICATION TO MIXTURE PROPORTIONING

The goal of the NIST PHPCT program is "To enable the reliable application of high-performance concrete in buildings and the civil infrastructure by developing, demonstrating, and providing assistance in implementing a computer-integrated knowledge system, HYPERCON, incorporating verified multi-attribute models for prediction and optimization of the performance and life-cycle cost of HPC." Substantial progress towards this goal has been made and the growing suite of materials science-based models that has already been developed for that purpose is now available on the web in the form of an "electronic monograph" [9]. Among the models are ones for predicting a) microstructure development in hardening cement paste from information on the composition, sizes and shapes of cement particles[10]; b) the structures of mortars and concretes from which transport properties can be calculated [11]; and c) the service life of chloride-exposed steel-reinforced concrete [12]. Each of the existing models will be refined as new insights are gained into the phenomena they represent, and complementary models to complete the set needed for optimization of mixture proportions for specific applications, such as models for the flow of concentrated particulate dispersions[13] and for fire response of fiber-containing concretes [14], are being developed. When the models are combined into interoperable computer-integrated knowledge systems available on the internet, they will provide a coherent materials science base for many aspects of concrete technology.

As mechanistic computer models improve, it may one day be possible to predict concrete performance through computer simulation prior to batching any concrete. Although this idealization is well into the future, current, and near future, mechanistic and microstructural computer models, such as those being developed at NIST, may one day be used to develop mixture proportioning guidelines. Over small regions of parameter space, nearly any function can be approximated sufficiently well by a quadratic model. However, for mixture proportioning guidelines to be useful for the concrete industry, the model would have to accommodate a large parameter space. Over these wide variations in parameters, mechanistic models can give insight to the optimum functional form for the model equation.

The combined use of computer models and factorial experiment design for estimating the diffusion coefficient of concrete has already been demonstrated [11]. A combination of composite theory and the NIST microstructural model for cement paste hydration was used to calculate the chloride diffusivities for concrete mixtures. The results from the calculations were used to determine the parameters of the model equation. The resulting estimates of diffusivity were demonstrated to be "not unreasonable" by comparison to reported data. One could imagine expanding this to other properties. The near future may hold similar experiments for concrete properties such as viscosity and yield stress. Numerical techniques for simulation of dense solid suspensions have been used to estimate the viscosity of a matrix containing solid particles [13]. Further developments may include estimates of yield stress. Results from these simulations could then be compared to experimental measurements using equipment that has been developed only recently [15,16].

These statistical techniques may also prove to be powerful tools for future concrete mixture design and optimization. Future optimizations will benefit from reasonably accurate trial mixtures based upon computer models and existing model equations. One could imagine a future ACI 211 composed of an analytical equation that gives not only a prediction for the best trial mixture, but also an estimate of the uncertainty of the response. This estimated uncertainty could then be used to establish the required parameter space for the optimization using zero trial mixtures. One could then bypass the step of making a series of trial mixtures that are currently required for establishing a reasonable parameter space.

SUMMARY

As awareness of the potential of concrete to achieve higher performance grows, the problem of designing concrete to exploit the possibilities becomes more complex. Statistical design of experiments, such as the response surface approach described in the present paper, is a tool than can be of immediate help in increasing efficiency in selecting the optimum proportions for a high-performance concrete. For the future, prediction of performance of concrete using materials science-based models in conjunction with standardized databases of concrete material property data should make possible multi-attribute optimization. The optimization should be able to take into account several attributes at a time, including flow properties, strength development, dimensional changes, fire response, life-cycle cost, and environmental impact.

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