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## Ternary mix design of grout material for structural repair using statistical tools

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### HIGHLIGHTS

- Sixteen grout mixtures containing three supplementary cementitious materials (SCMs) are formulated.
- Rheological, shrinkage and mechanical properties are determined.
- Statistical models are formulated to characterize the effects of various SCMs on properties investigated.
- Properties are evaluated for structural performance leading to optimization of SCMs addition.

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### ABSTRACT

Repair is an indispensable part of the maintenance of structures over their lifetimes. Structural grouting is a widely used remediation technique for concrete components, trenches, mine subsidence, dam joints, restoration of masonry structures, and geological stabilizations. A structural grout system should be injectable in narrow spaces and hence include ingredients with finer particles. Ultrafine cements are ideal for these type of demanding grouts due to their superior properties compared to that of the less-expensive, but coarser ordinary Portland cement (OPC). Supplementary cementitious materials (SCMs) are often used to replace OPC clinker based binder in order to modify certain properties and to reduce costs. The most commonly used SCMs are fly ash (FA), and ground granulated blast furnace slag (GGBS). For various special applications microsilica (MS), and metakaolin (MK) are also used. Identifying the optimum replacement contents of OPC by SCMs are a challenge during the design of such grouts. The aim of this experimental study is to investigate the effect of the selected SCMs (FA, MS and MK) on the slump flow, time of efflux, viscosity, shrinkage, and compressive and flexural strength of ultrafine cement based grouts with constant water-binder ratio and superplasticizer content. The test program was formulated using Box-Behnken design principles. Maximum percentages of replacement with ultrafine cement was 6% by volume of cement for MS and 16% for FA, and MK. The results suggest that most investigated grouts have the potential to be used for structural applications. The appropriate quadratic models are then formulated through statistical tools and presented as response surfaces. The trends indicate that fly ash improves the rheological properties, whereas microsilica and metakaolin positively affect shrinkage and mechanical properties to some extent. Based on the influence of SCMs and priorities among the properties, Decision Matrix Analysis (DMA) is carried out to select the most suitable ones among the SCMs. The analysis suggests that microsilica and fly ash are more suitable as SCMs than metakaolin without affecting the properties.

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### 1. Introduction

Grouts are ideal materials for repair of concrete and composite structures. Besides, grouts are widely used for tunneling, soil stabilization, and foundation technologies both on onshore and offshore

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engineering sites. Repair is even more critical for remote offshore energy structures, where advanced technical expertise and higher financial costs are involved. Application of such products poses considerable challenges due to their specific properties during mixing, pumping, spreading or injection, and service conditions. The rheological properties and stability are thus important in determining the penetrability of such cement grouts [1]. A higher water content usually provides certain favorable rheological

properties, but may, however, not be suitable for some other requirements and thus requires optimization [2–5]. A combination of suitable ingredients is necessary to achieve those desired properties.

Generally, there are two most commonly sorted grout systems: cement based grouts and chemical grouts, which are defined by the type of binders used in them. Chemical grouts are primarily epoxy-based, polyurethane-based, and other polymer grouts. A cement based grout is sometimes preferred due to compatibility with the substrate and environmental superiority. However, finer cement is required to achieve better penetrability. Ultrafine cements are ideal for structural applications due to superior permeability and strength properties compared to that of the less expensive, but coarser OPCs [6,7]. This type of cement has an extremely small particle size with a maximum particle size of only few microns. The interaction of these finer particles affect properties of the final product [8,9]. Most of these ultrafine cements are quite similar in their properties as conventional OPCs. When compared to chemical grouts, ultrafine cement grouts offer an economical and efficient alternative. The financial advantage can further be enhanced when cement is partially replaced with supplementary cementitious materials (SCMs). A structural grout should be suitable for pumping and physically adequate as structural filler at hardened stage. For this reason, coarser aggregate should be avoided. Microsilica (MS), fly ash (FA), bentonite (B), granulated slag (GGBS), and meta-kaolin (MK) are the commonly used pozzolanic materials for special grout applications [10–14]. However, their inclusions affect various properties. Besides, plasticizing admixture is typically necessary to attain the required properties [11,15]. Since two or more variables are involved, a parametric study is essential to attain suitable properties from optimum contents of SCMs.

This study is aimed at determining the range of ingredients that can produce a structural grout using ultrafine cement as primary binder. Slump flow, time of efflux, and viscosity were determined as an indication of rheological properties. Whereas compressive and flexural strengths were determined to assess physical properties. The observation of volumetric and linear shrinkage was also included in this study. A statistical mix design approach was used and results are presented using ternary response surfaces. Mathematical models were formulated for all properties involved. Established models were verified using statistical analysis of variance. It also shows how statistical models, which are quadratic in nature, can be used to develop tools for future predictions. The general trends of inclusion of the supplementary materials on each property are identified and discussed. Finally, grout systems with optimum ingredients suitable for structural repair are proposed.

## 2. Experimental program

### 2.1. Experimental design and analysis techniques

The Design of Experiments (DOE) is an efficient statistical procedure for planning experiments so that a series of data obtained can be analysed to obtain valid optimum parameters. This procedure uses the experimental data to develop an empirical model linking outputs and inputs. The simplest DOE can be one factor design, where one parameter is changed and other are kept constant. However, in case of larger variables, this process can result in an increased number of tests, which eventually increases testing and decision time along with associated cost. A Central Composite Design (CCD) contains an imbedded factorial or fractional factorial design with centre points associated with a set of high and low boundary points allowing estimation of curvature. If the distance from the centre of the design space to a factorial point is  $\pm 1$  unit, the distance from the centre of the design space to any boundary

point is  $|\alpha| > 1$ . The precise value of  $\alpha$  depends on certain properties desired for the design and on the number of factors involved. Being first devised in 1960, Box-Behnken is one of the experimental design techniques that investigates the influence of different variables on the outcome of a controlled experiment using experimental runs in blocks within coded unit values of  $-1$ ,  $0$ , and  $1$  without extending additional boundary points for  $\alpha$  [16].

These blocks are connected with pivoting tests with coded units of  $0$ ,  $0$ , and  $0$ . Three-level incomplete factorials are used in this study of quantitative variables. This Box-Behnken design methodology is adopted over typical CCD due to the fact that it requires less test runs to observe the trend in output with reasonable statistical accuracy. Being a nearly rotatable design with three evenly spaced levels, Box-Behnken can also be used to identify the individual and interaction effects when mathematical models are established.

The second degree graduating polynomial that best represents the relation is given as Eq. (1), where  $x_i$  refers to the input factors which influence the response  $y$ . The first coefficient  $a_0$  denotes the mean, and  $\epsilon_x$  is the random error. The parameter  $a_i$  represents individual effect,  $a_{ii}$  represents the quadratic effect of the  $i$ th factor, and  $a_{ij}$  represents the interaction between the  $i$ th and  $j$ th factors. Eq. (1) can be broken down to Eq. (2), where the combined coefficient,  $a_x = a_0 + \epsilon_x$  represents the combination of the mean and the provision for an error adjustment.  $x_1$ ,  $x_2$ , and  $x_3$  are MS, FA, and MK contents, which are variables of interests in this study.

$$y = a_0 + \sum_{i=1}^n a_i x_i + \sum_{i=1}^n a_{ii} x_i^2 + \sum_{i < j} a_{ij} x_i x_j + \epsilon_x \quad (1)$$

$$y = a_x + a_1 x_1 + a_2 x_2 + a_3 x_3 + a_4 x_1^2 + a_5 x_2^2 + a_6 x_3^2 + a_7 x_1 x_2 + a_8 x_1 x_3 + a_9 x_2 x_3 \quad (2)$$

A total 15 test runs are required for three factors considered in this study. The contents correspond to coded value specified by [16]. Table 1 shows the test matrix and ingredients. Qualitatively, blending of cement with silica will lead to a decrease of the amount of portlandite and the formation of more CSH with a lower Ca/Si ratio [17]. For this reason, priorities were given to MS, FA, and MK, whose contents are kept to 0–6%, 0–16%, and 0–16%, respectively. Moreover, these contents were chosen from an overview of previous literature, where comparable rheological and mechanical properties were expected. MS was chosen after [18,19], where maximum 5–8% by mass replacement was adopted. A FA content of maximum 20% by mass was used [20]. Finally, a MK content range up to 16% was chosen after [5], where 6–20% was used. It can be seen that  $-1$ ,  $0$ , and  $+1$  represent the corresponding lowest, medium, and maximum values, respectively. Cement contents were replaced with SCM contents by volume. An additional mixture number 16 is shown, which represents the mixture with neat cement.

Once results are obtained for the designed experimental program, solution of response equations was carried out using the SOLVER tool of the Microsoft Excel with an aim to achieve Goodness of Fit by minimizing the *chi square*;  $\sum (\chi)^2 = \sum \frac{(\text{Observed} - \text{Estimated})^2}{\text{Estimated}}$ . The process itself is a regression analysis. The reason for using these analogy was to achieve the best fit predictions, since errors were minimized in comparison to individual data points.

A common way of representing experimental design outcomes is the Response Surface Methodology (RSM) [21]. RSM provides an ease of observations among the variables and combined effects spanning the entire experimental region of interest. This method was actually developed by Box and Wilson in 1951 to aid the improvement of manufacturing processes in the chemical industry

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