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Optimisation of rheological parameters and mechanical properties of superplasticised cement grouts containing metakaolin and viscosity modifying admixture

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HIGHLIGHTS

- ▶ Percentage of SP, VMA, and MTK had a significant effect on the fluidity, rheology, and setting times.
- ▶ MTK significantly reduced setting times and improved the compressive strength at 3 d, 7 d, and 28 d.
- ▶ The desirability function increased with an increase in % MTK and tended to reduce when SP increased.

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ABSTRACT

The objective of this research was to optimise the rheological parameters, hardened properties, and setting times of cement grouts containing metakaolin (MTK), viscosity-modifying agent (VMA) and superplasticiser (SP). All mixes were made with water-to-binder ratio (W/B) of 0.40. The replacement of cement by MTK was varied from 6% to 20% (by mass), and dosages of SP and VMA were varied from 0.3% to 1.4%, and 0.01% and 0.06% (by mass of binder), respectively. Increased SP led to an increase in fluidity, reduction in flow time, plate cohesion, rheological parameters, and an increase in the setting times. Increased VMA demonstrated a reduction in fluidity, an increase in Marsh cone time, plate cohesion, yield stress, and plastic viscosity. Results indicate that the use of MTK increased yield stress, plastic viscosity, cohesion plate, and flow time due to the higher surface area associated with an increase in the water demand. MTK reduced mini-slump and setting times, and improved compressive strength.

1. Introduction

Grouting is used to fill and bond cracks and defects in structural concrete and masonry; grout has to be highly flowable, with a maximum particle size considerably smaller than the thickness of the width of cracks [1]. Cementitious grouts are being used for an increasing number of exciting structural purposes, and are able to withstand the increasing demands being placed on them. Many of these developments have been made possible through the use of chemical admixtures and cement replacement materials, such as pulverised fly ash (PFA), ground granulated blast furnace slag, silica fume, metakaolin (MTK) and limestone powder, which are now widely accepted as some of these cementitious materials having economic aspect and, more critically, technical advantages in all cementitious materials [2–10].

Viscosity modifying admixtures (VMAs) have proved to be very effective in stabilizing rheological properties and fluidity of cement grout, self-compacting concrete, and underwater concrete [7,8,11–13]. Recently, there has been a growing interest in the use of cement grout containing viscosity admixture for repairs, injection, embedding of anchors and post-tensioning, and rock and oil-well grouting [14,15]. A cement-based grout should be stable enough to reduce sedimentation, bleeding, and water dilution. Cement-based grouts are widely used in injection grouting of cracks in massive structures since their physical and mechanical properties can be easily controlled. This control is assured by judicious choice of cement type and fineness, water-to-binder ratio, and chemical and mineral admixtures [1]. Incorporating one or more types of supplementary cement replacement materials (CRMs) [8,9,12] such as PFA, which has different morphology and grain-size distribution values can improve particle size distribution and packing of solid particles, enhance fluidity, improve stability [8] and reduce permeability. Improvements in the quality and uniformity of CRMs

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and the attention being given to admixture formulations have also assisted progress.

VMAs are used to enhance the cohesion and stability of cement-based systems. They are generally water-soluble polysaccharides, synthetic or semi-synthetics that enhance the water retention capacity of paste [4,8,16]. The use of VMA increases yield stress and plastic viscosity of cement-based grout [7,8,11,13], thus necessitating an increase in water-to-binder ratio (W/B) or superplasticizer (SP) dosage to ensure the low yield stress necessary for proper penetrability and spreading in cracks [5,7,8]. Increased W/B reduces mechanical properties and durability, and increases permeability [1,5].

The main objective of this study was to evaluate the effect of MTK, SP and VMA dosage on the grout fluidity, rheological properties, setting times, and compressive strength at 3, 7 and 28 days using factorial design approach and analysis. Simulation of models obtained with factorial design can facilitate the test protocol needed to optimise cement grout within a given set of performance criteria. Multi-parametric optimisation (using the desirability function) was used to establish response surfaces.

2. Research significance

Optimisation of cement grouts often requires several trial batches to achieve an adequate balance between rheological properties and mechanical performance. A factorial design approach was used to determine the effect of mineral and chemical admixtures such as MTK, SP and VMA on the fluidity, rheological parameters, setting times, and compressive strengths of cement grouts. The optimum balance among various mix variables was obtained by minimising the addition of SP and VMA to maximise fluidity, viscosity, and mechanical performance and minimise yield stress and setting time, and produce a stable cement grouts with adequate rheological parameters.

3. Statistical design of experiments

Central composite experimental design (CCED) was the analytical technique used to evaluate the influence of maximum and minimum levels of each variable on relevant grout properties [17]. CCED was selected to limit the total number of cement grouts investigated and to allow first-and second order models to be used to fit the experimental data. In this study, three key parameters which can influence the mix characteristics of cement grout were selected to use in formulating mathematical models for evaluating relevant properties. The minimum and maximum experimental levels of MTK, SP and VMA are defined in Table 1. These levels were selected based on some preliminary results and also the lowest limits of VMA, SP using CCED were almost 0%.

This type of two-level fractional factorial design requires a minimum number of tests for each variable. Since expected responses do not vary in a linear manner with the selected variable, a CCED was selected to enable quantification of the response prediction where the response could be modelled in a quadratic manner. Since the level of error in predicting responses increases with the distance from the centre of the modelled region, it is advisable to limit the use of models to an area bound by coded values corresponding to $-\alpha$ to $+\alpha$ limits. The α value is chosen so that the variance of the response predicted by the model would depend only on the distance from the centre of the modelled region. CCED is made rotatable by the choice of α, which depends on the number of mixes in the factorial design. In fact, $\alpha = (n_F)^{1/4}$ yields a rotatable CCED where n_F is the number used in the factorial portion of the design ($n_F = 8$). For the experimental design in this study, the two values of α are ±1.41.

The modelled experimental region consisted of mixes with coded variables ranging from −1.41 to +1.41. The factorial models are valid for mixes made with 6–20% MTK, 0.3–1.4% SP by mass of binder, and concentrations of 0.01% to 0.06% VMA by mass of binder. The responses modelled were mini-slump, Marsh cone, plate cohesion, yield stress, plastic viscosity, initial and final setting times, and 3-d, 7-d and 28-d compressive strengths.

Fifteen mixes were considered in the experimental design of cement grouts. Due to the fact that two-level fractional factorial design does not allow for an estimate of the experimental error unless some runs are repeated five replicate central points were prepared to estimate the degree of experimental error for the modelled responses. The central points consisted of mixes where variables were fixed in the middle level corresponding to 13% MTK, 0.85% SP, and 0.035% VMA. In total, twenty mix combinations were tested with an extra five mixes as verification points for accuracy (Table 2).

Table 1
Coded and absolute values for the investigated parameters.

Parameter	−1.41	−1	Central point	+1	+1.41
MTK	3.1	6	13	20	22.9
SP (%)	0	0.3	0.85	1.4	1.63
VMA	0	0.01	0.035	0.06	0.07

Table 2
Mix proportions of all mixes tested in this study.

	Coded values				Absolute values			
	Mix	MTK	SP	VMA	MTK	SP	VMA	W/B
Level of factors	1	1.00	1.00	−1.00	20.0	1.40	0.010	0.40
	2	1.00	−1.00	1.00	20.0	0.30	0.060	0.40
	3	−1.00	1.00	1.00	6.0	1.40	0.060	0.40
	4	−1.00	−1.00	−1.00	6.0	0.30	0.010	0.40
	5	−1.41	0.00	0.00	3.1	0.85	0.035	0.40
	6	1.41	0.00	0.00	22.9	0.85	0.035	0.40
	7	0.00	−1.41	0.00	13.0	0.07	0.035	0.40
	8	0.00	1.41	0.00	13.0	1.63	0.035	0.40
	9	0.00	0.00	−1.41	13.0	0.85	0.000	0.40
	10	0.00	0.00	1.41	13.0	0.85	0.070	0.40
Centre points	11	0.00	0.00	0.00	13.0	0.85	0.035	0.40
	12	0.00	0.00	0.00	13.0	0.85	0.035	0.40
	13	0.00	0.00	0.00	13.0	0.85	0.035	0.40
	14	0.00	0.00	0.00	13.0	0.85	0.035	0.40
	15	0.00	0.00	0.00	13.0	0.85	0.035	0.40
Points of verification	16	−1.20	−0.50	0.00	4.6	0.58	0.035	0.40
	17	−1.30	−0.40	0.00	3.9	0.63	0.035	0.40
	18	0.50	0.00	0.00	16.5	0.85	0.035	0.40
	19	1.00	0.60	1.00	20.0	1.18	0.060	0.40
	20	0.80	0.40	0.80	18.6	1.07	0.055	0.40

4. Materials properties and mix proportions

The grout mixes investigated in this study were prepared with Standard CEM I 42.5N Portland cement (PC) and MTK. MTK is a highly processed form of kaolin clay, which is a mineralogically complex hydrous aluminium silicate, white in colour with a dull lustre. It is a processed material, calcined in kilns to form highly reactive material known as MTK. MTK is a pozzolan, which is a material that, when mixed with water will chemically react with calcium hydroxide at normal temperatures to form compounds possessing cementitious properties. The chemical composition of CEM I and MTK are provided in Table 3. XRD analysis results of cement and metakaolin were presented in Fig. 1. The appearance of hump (between $2\theta = 20^\circ$ and 30°) indicates the formation of an