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Optimization of Cement-Silica Fume Blends for The Suppression of Sulphate Soil Swelling

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تحديد نسبة الخلط الأمثل للأسمنت وغبار السيليكا لقمع انتفاخ التربة الكبريتية

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Abstract:
The expansion resulting from the ettringite nucleation in sulfate soil treated with calcium-based stabilisers causes a technical and economic issue for geotechnical engineering. Thus, this research investigates the practicability of co-utilization of cement (C) and silica fume (S) at a variety of blending ratios (10, 30, 50 and 70 wt%), with a view of determining the optimum blinder of C–S for restricting the development of ettringite minerals in soil samples rich in sulfate (9% gypsum). To achieve this target, a series of laboratory samples, prepared at different cement and silica fume concentrations, were examined using unconfined compression strength (UCS), linear expansion, and derivative thermo-gravimetric analysis (DTG) as the key criteria for the evaluation. The research observations revealed a gradual reduction in strength and expansion, as the substitution ratio of cement with silica fume increased to 30% and 70%, respectively. Therefore, 7C–3S was categorised as the optimal blending ratio for UCS, while 7C–3S was superior in terms of the expansion, as it exhibited a lower expansion magnitude of 1.42%. Accordingly, the use of silica fume has proven to be an effective approach for restricting the nucleation of ettringite minerals in cemented sulfate-bearing soil.

Keywords: Sulfate Bearing-Soil, Ettringite, Calcium-Based Stabiliser, Cement, Silica Fume, Strength, Linear Expansion, Swelling.

الملخص
يُسبب التمدد الناتج عن تكوين نوى الإترينجيت في تربة كبريتية مُعالجة بمثبتات أساسها الكالسيوم مشكلة تقنية واقتصادية في مجال الهندسة الجيوتقنية. لذا، يبحث هذا البحث في جدوى الاستخدام المشترك للإسمنت وغبار السيليكا بنسب خلط مُتنوعة (10، 30، 50 و70%)، بهدف تحديد النسبة المثلى للحد من نمو معادن الإترينجيت في عينات تربة غنية بالكبريتات (9% جبس). ولتحقيق هذا الهدف، فُحصت سلسلة من العينات المخبرية، المُحضرة بتركيزات مُختلفة من الإسمنت وغبار السيليكا، باستخدام قوة الضغط غير المُقيدة، والتمدد الخطي، والتحليل الحراري الوزني المُشتق كمعايير

رئيسية للتقييم. كشفت ملاحظات البحث عن انخفاض تدريجي في القوة والتمدد، مع زيادة نسبة استبدال الإسمنت بغبار السيليكا إلى 30% و70% على التوالي. لذلك، صُنِّفت نسبة (70% اسمنت و30% سيليكا) كنسبة المزج الأمثل لقوة الضغط غير المحصورة، بينما تفوقت نسبة (30% اسمنت و70% سيليكا) من حيث التمدد، حيث أظهرت نسبة تمدد أقل بلغت 1.42%. وبناءً على ذلك، أثبت استخدام دخان السيليكا فعاليته في الحد من تكوين معادن الإترينجيت في التربة الأسمنتية الحاملة للكبريتات.

الكلمات المفتاحية: تربة حاملة للكبريتات، إترينجيت، مثبت أساسه الكالسيوم، أسمنت، سيليكا، قوة، تمدد خطي، انتفاخ.

Introduction

Expansive clays, especially those containing high sulfate content, are encountered in about 20% of the worldwide [1–3], and categorised as a problematic soil in the presence of calcium-based binders such as Portland cement and lime [4–7]. This problematic phenomenon is traditionally attributed to the heaving [8–12], caused by the nucleation of expansive minerals, i.e., ettringite minerals [13]. The ettringite minerals are highly crystalline minerals formed through the reaction between calcium, aluminium, and sulfate in the presence of moisture [14]. Ettringite minerals also have a highly water-holding capacity, intensive expansion behaviour and fast growth nature [6,15], the growth of which induces a drastic expansion of the soil profile, and threatens the sustainability of engineering structures such as pavements and roads [13,16]. Consequently, several research studies have been conducted to evaluate the understanding of the mechanism of ettringite formation and its effect on soil stabilisation with calcium-based stabilisers. In this context, Celik and Nailbantoglu [17] concluded that the lime stabilisation technique is effective for stabilisation of sulfate soil containing up to 2% sulfate concentration. Aldaood, Bouasker, and Al-Mukhtar [18] reported that calcium sulfate concentration, beyond which the unconfined compressive strength performance is decreased, was about 5%. Similarly, Jha and Sivapullaiah [19] reported a drastic increase in swelling of lime-stabilised expansive soil as the sulfate content increases, and such a swelling increase was more pronounced at higher sulfate content (above 6%).

The manufacturing of cement is also of great influence on the economy, as it requires a huge amount of energy, about 5000 MJ/tonne for cement production, accounting for about 2% of global energy [16]. Cement production also releases massive carbon dioxide emissions of 1000 kg/tonne per tonne and consumes intensive raw materials (1.5 tonnes of clay per one tonne of cement) [20], of which the latter causes the depletion of natural raw materials and threatens sustainability. Therefore, the use of low calcium-based binders (such as pulverised fly ash) and industrial byproducts rich in aluminosilicate compounds (GGBS and silica fume) as a partial substitution of calcium-based stabiliser is encouraged [16,21]. In this regard, Li, Fu, Xiao, Xi and Yang [22] examined the impact of different sulfate types and concentration on the performance of different soils stabilised with a binary blend of lime-GGBS, and reported a variation in soil performance with the change of sulfate type and concentration. Seco, Ramírez, Miqueleiz, and García [23] studied the effect of binary combination of 20% lime and 20% rice husk ash on the performance of expansive soil. The observation of such a study exhibited an 800% strength enhancement, 90% plasticity reduction, and 70% swelling decrease, due to the modification of fabric and the formation of pozzolanic reactions [6,24].

As an alternative pozzolanic silica fume, a by-product of silicon alloy industries, has also gained great attention in the field of sulfate soil stabilisation [13,20,25–28]. To name a few, a research study of [29] reported a reduction (from 6% to about 0%) in swelling of sulfate soil by using a ternary combination of cement, GGBS and silica fume. A study conducted by [30] examined the effect of comprehensive combinations of lime and silica fume on the expansion of high-sulfate silty sand and reported a considerable swelling reduction and strength increase of up to 12 times the original soil. A study conducted by [31] investigated the impact of nano-silica on the characterisation of expansive soil and achieved the optimal compressive strength by using a combination of 1.2% of nano-alumina and 2% of nano-silica. Mousavi [32] also investigated the effect of co-addition of cement and silica fume on the performance of high plastic clay having a sulfate content of 2.7% and observed that 6% cement and 2% silica fume was the optimal blend in terms of the shear strength, with no reported investigation on swelling. Accordingly, the main conclusion that can be drawn from the above-mentioned studies is that Silica fume addition in sulfate soil is an effective method due to its characteristics, such as its richness in silicon dioxide and its higher pozzolanicity. These characteristics accelerate the consumption of calcium hydroxide and form additional hydrates, preventing the formation of ettringite [20,33], thus increasing the strength and reducing swelling potential effectively [31,34]. However, a research study considering the addition of a wide range of cement and silica fume combinations in sulfate, in order to establish the threshold of cement/silica fume ratio at which the swelling of sulfate soil is suppressed, cannot be found in the literature.

This research study, therefore, aimed at the optimisation of various combinations of cement and silica fume at a fixed binder content of 10% for sulfate soil stabilisation. To do so, a series of stabilised specimens were investigated by using unconfined compressive strength, linear expansion, and derivative thermogravimetric analyses.

Material and methods

Three raw materials, with oxide compositions presented in Table 1, physical compositions tabulated in Table 2, particle size distribution shown in Figure 1 and DTG curve illustrated in Figures 2 and 3, were utilised throughout the laboratory experimentations. These materials were:

- Industrial kaolin in the form of white odourless powder, having a relative density of 2.6-2.7. It was supplied by Potterycrafts Ltd, England, under the commercial trade name of China Clay Standard Porcelain Powder. It was used in this study due to the consistency and homogeneity [35], as it facilitates the ease of illustrating and recognising some complex interactions before proceeding into more complex natural untreated clay soils in future work [20];
- Calcium sulfate dihydrate (Gypsum) in the form of a fine white powder, having a percentage purity of $\geq 98\%$ and water solubility of 2 g/L (20℃). It was obtained from Fisher Scientific Ltd, Loughborough, Leicestershire, England, UK;
- Portland cement in the form of a fine grey powder, having a water solubility of 1337.6 mg/L, specific gravity of 3.15 Mg/m^3, bulk density of 1400 kg/m^3 and alkalinity value (pH value) of 13.41. It was supplied under the commercial trade name of Premium Cement from Premier Cement Limited, UK); and
- Silica fume in the form of a fine, grey amorphous powder containing a silicon dioxide content of 98.4%. It was originally produced by Elkem Silicon Materials in Norway, and acquired from Tarmac Cement and Lime Company, Buxton lime and powders, Derbyshire, Derby, UK, under a commercial brand of undensified silica fume.

Table 1: Oxide composition of kaolin, Portland cement, and silica fume.

Oxides	Compositions (%)		
	Kaolin	Cement	Silica fume
CaO	<0.01	61.49	0.2
MgO	0.21	3.54	0.1
SiO_2	47.32	18.84	98.4
Al_2O_3	35.96	4.77	0.2
Na_2O	0.07	0.02	-
P_2O_5	0.12	0.1	0.03
Fe_2O_3	0.69	2.87	0.01
Mn_2O_3	0.02	0.05	-
K_2O	1.8	0.57	0.2
TiO_2	0.02	0.26	-
V_2O_5	<0.01	0.06	-
BaO	0.07	0.05	-
SO_3	0.01	3.12	0.1
LOI	13.1	4.3	0.5

Table 2: Some physical properties for the kaolin, Portland cement, and silica fume.

Properties	Kaolin	Portland cement	Silica fume
Linear Shrinkage (%)	10.8	-	-
Linear Expansion (%)	6.2	-	-
Swelling Pressure (kPa)	1.3	-	-
Bulk density (kg/m^3)	-	1400	300
Specific gravity (Mg/m^3)	2.14	3.15	3.15
Alkalinity value (pH)	5.37	13.41	7
Colour	White	Grey	Grey

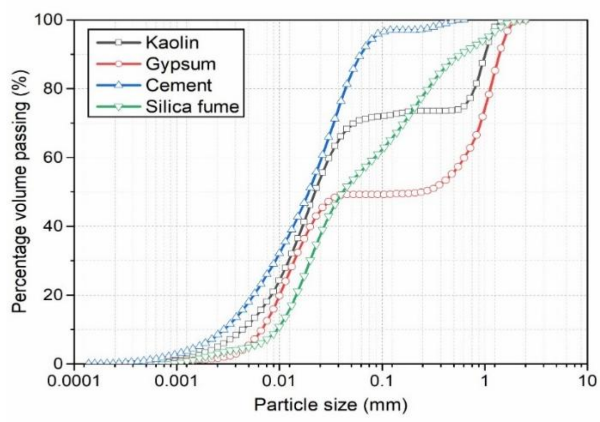

Figure 1: Particle size distribution of kaolin, gypsum, cement, and silica fume.

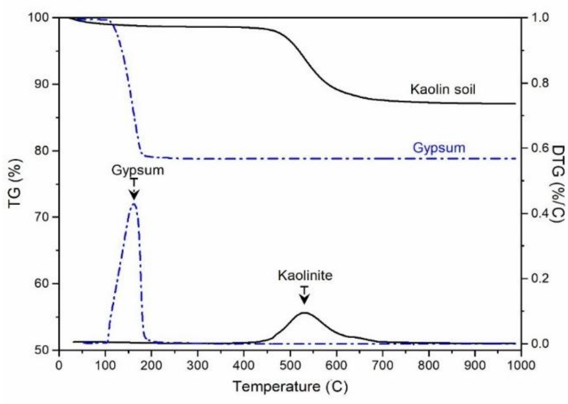

Figure 2: TG/DTG curves of Kaolin and Gypsum.

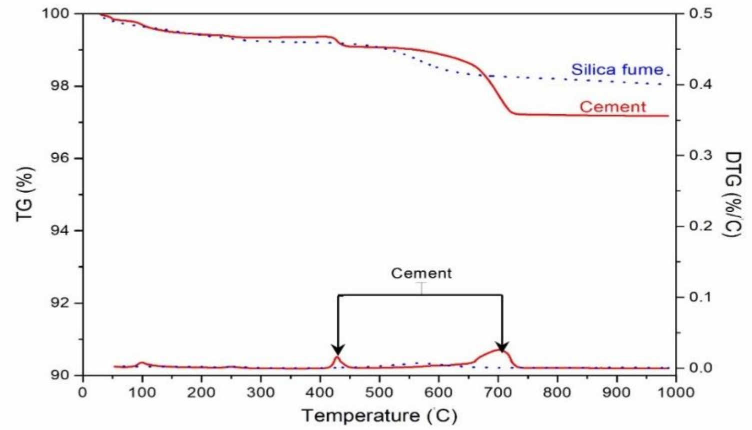

Figure 3: TG/DTG curves of Cement and Silica Fume.

Mix composition

Different mix compositions were designed under this study, as seen in Table 3, to establish the silica fume threshold beyond which further increase of silica fume has no significant contribution to the suppression of cement-stabilised sulfate soil expansion. These mix compositions were formed by mass substitutions of cement with silica fume at a substitution level of 10%, 30%, 50% to 70%, respectively. A mix designation code comprised of two parts; the first part refers to the artificial sulfate soil (K9G), while the remained part refers to the binder composition, of which C (Cement), and S (Silica fume) were preceded by a number representing the binding material percentage.

Table 3: Mix design using pure kaolin and artificial sulfate-dosed kaolin.

Mix Code	Mix compositions (%)					
	Target Soil Materials (%)		Water (%)	Binder (%)	Binder composition in % by Target Soil Material mass	
	Kaolin	Gypsum			Portland cement	Silica fume
K0G–10C0S	100	0	31	10	10	-
K9G–10C0S	91	9	31	10	10	-
K9G–9C1S	91	9	31	10	9	1
K9G–7C3S	91	9	31	10	7	3
K9G–5C5S	91	9	31	10	5	5

Sample preparation

A total of 12 samples per mix composition were prepared by mixing the solid ingredients (kaolin, sulfate, cement, and silica fume) with a moisture content equal to the optimum moisture content (OMC) of 31%. For each sample, the solid materials were mixed using a mechanical mixer device for 3 min before the predetermined OMC of 31% was introduced gradually. On achieving a homogenous mixture, the semi-paste admixture was carefully placed into a pre-lubricated steel mould measuring 100 mm in length and 50 mm in diameter. The homogenised admixture was then pressed using a hydraulic jack wherein a static compressive force was axially applied in aid of a fabricated custom-built steel frame, to obtain a specimen with dimensions of 100 mm in height and 50 mm in diameter. The sample was then cautiously extracted employing a steel cylinder plunger lubricated in advance to ease the extraction process. Thereafter, the prepared samples were wrapped in cling film to reduce moisture evaporation. Finally, the produced samples were cured in a plastic container at a temperature-controlled room of 20 ◦C until the date of testing.

Testing method

Three tests, including UCS, linear expansion, and TG/DTG analysis, were conducted in this study to assess the performance of the designed samples.

Linear expansion

The swelling test was performed in adherence with [36], by using Perspex cells such that used elsewhere [37–39]. Three specimens per mix proportion were used during the analysis to ensure the accuracy/reliability of the outcomes [21]. Directly after 7 days of curing, about 10 mm-bottom of the cling film of the three cylindrical samples was removed using a sharp razor. The partially exposed samples were then individually installed on a separate porous disc, located in a plastic platform in a Perspex cell. The Perspex cells were thereafter covered with prefabricated lids, fitted with dial gauges to measure the vertical expansion of the samples. The water was carefully introduced until the uncovered part of the samples was completely immersed in water (up to 10 mm of the samples' base), and the dial gauge's reading was recorded on a weekly basis, until no change in reading was observed. Finally, the linear expansion magnitude was used to establish the linear expansion of the samples, i.e., the ratio of change in height to the original height before expansion.

Unconfined compression strength

The UCS was carried out in adherence with [40], by using a Hounsfield testing machine equipped with a special self-levelling device to ensure uniaxial load application. At the end of each curing period (7, 28, and 90 days), the cling film of three cylindrical samples was removed, and the samples were subjected to a uniform compressive load at a constant strain rate of 2 mm per minute. Thereafter, the average of the failure loads was used to determine the unconfined compressive strength and reported in this study as the representative unconfined compressive strength.

Mereological and analytical tests

The DTG analysis was conducted on the pre-dried pre-powdered portion collected from the middle of the broken UCS samples at the 7-day curing. The testing was performed from room temperatures up to 1000 °C, under an argon atmosphere, and at a flow heating rate of 20 °C per minute, using a TA Instruments TGA55 kit.

Results and discussion

Unconfined Compressive Strength (UCS)

The unconfined compressive strength of sulfate kaolin specimens stabilised with different proportions of cement and silica fume at 7, 28 and 90 days of moist curing is illustrated in Figure 4. In general, all the stabilised specimens exhibited a gradual increase in strength corresponding to the increase in age of curing. This is typically attributed to the fabric modification through cation exchange and soil particle agglomeration, and the formation of an interlocked matrix through the formation of hydrated products (pozzolanic reactions). Upon the substitution of cement with silica fume, the results revealed a gradual increase in strength up to a 30 % substitution level, in which the strength increased from 2030 kN/m^2 to 2090 kN/m^2, as represented by 7C-3S. This substitution level indicates the optimal performance of strength, probably due to the complete consumption of calcium hydroxide produced through cement hydration and the formation of future hydrates due to the pozzolanic reaction with silica fume [41]. The blockage of pores due to the filler effect of silica fume is also a possible contributing factor for the superior performance of the binary blend of 7C-3S, as it improves the porosity of the system, thereby increasing the roughness against loading.

Upon further substitution of cement with silica fume, however, there was a gradual reduction in strength, accounting for the lowest value of 890 kN/m^2 at a substitution level of 70%, as represented by 3C-7S. This deteriorative effect of silica fume can be attributed to the high pozzolanicity of silica fume and the lower content of cement in the system. According to Suraneni and Weiss [42,43], the undensified silica fume utilised in this study had a pozzolanic activity even higher than GGBS and calcined clay, where it can consume calcium hydroxide up to four times higher than GGBS. This, therefore, indicated that the undensified silica fume speeds up the consumption of $Ca\ (OH)_2$, thus restricting the formation of ettringite in the system, thereby, a lower strength enhancement associated with ettringite formation has occurred [41].

Linear Expansion

Figure 5 plots the observed expansion behaviour of sulfate soil specimens treated with a binary admixture of cement-silica fume through a prolonged soaking period of 200 days.

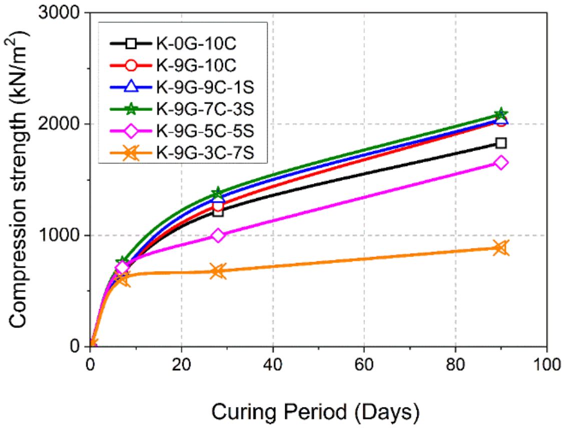

Figure 4: Representative unconfined compression strength development (up to 90 days) of cylindrical kaolin specimens stabilised using varying levels of Cement-Silica fume (CS) blends.

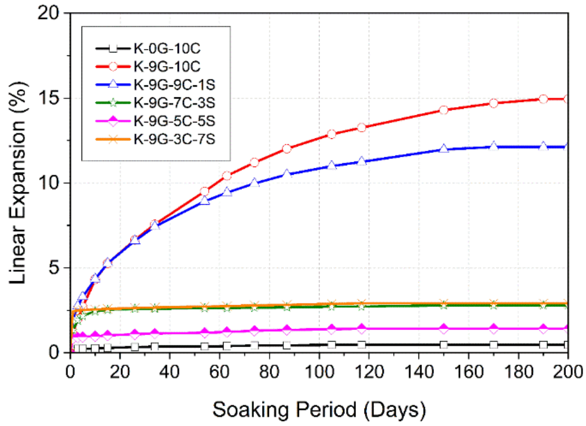

Figure 5: 200 days-Linear expansion behaviour of kaolin specimens containing 0 and 9% Gypsum-G and stabilised with binary blends of Cement-Silica fume (C-S).

As shown in the figure 5, the substitution of cement with silica fume played a significant role in enhancing the expansion behaviour of sulfate soil, and such an enhancement was more pronounced at relatively higher substitution levels of up to 70%. This was presented by a continuous decrease in expansion magnitude from 14.95% (for 10C) to about 1.42% (for 5C-5S), beyond which a slight increase in expansion to about 2.92% was observed, as represented by the 3C-7S mixture. The reduction in swelling values can be assigned to the inhibition of ettringite nucleation because of the higher pozzolanic activity of silica fume. The higher pozzolanic activity impacts the hydration process by promoting more active nucleation sites to accelerate the consumption of portlandite, thus reducing the solubility rate of alumina [7,44]. The inhibition of alumina dissolution restricts the ettringite production and enhances the production of CSH over that of CAH, all of which mitigates expansion [21]. As for the increase in expansion magnitude of higher cement substitution levels, this can be credited to the lower content of hydrated products responsible for the interlocking of the system due to the lower content of cement within the system.

Derivative-Thermos-Gravimetric analysis (DTG)

Figure 6 shows the thermal decompositions of sulfate kaolin specimens stabilised with different proportions of cement and silica fume at 7 days of moist curing age. In general, the main endothermic peaks detected in the DTG analyses were 1) the ettringite peak at a temperature range of 50 °C to 100 °C. 2) gypsum peak at a temperature range of 100 °C to 200 °C, portlandite peak at a temperature range of 400 °C to 500 °C, and kaolin peak at a temperature range of 450 °C to 700 °C [45].

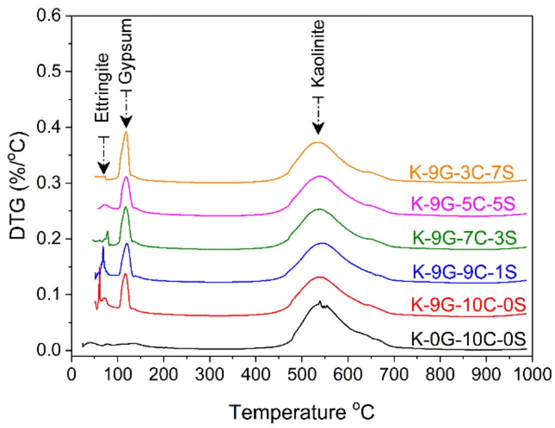

Figure 6: The 7th day-DTG curves of artificial kaolin specimens dosed with 0 and 9% of gypsum and stabilised with different combinations of cement and silica fume at a binder level of 10 wt. %.

By comparing the intensity of ettringite peak, it was observed that the intensity of ettringite peak decreased gradually as the cement substitution level increased, indicating the role of silica fume in restricting the formation of ettringite. This is therefore in partial support of why the strength was increased while the expansion was reduced on the substitution of cement with silica fume. This was also further validated by the gradual increase in the intensity of gypsum peak, which also indicates a sign of restriction of ettringite formation [41]. The ettringite formation, under moist curing, improves the strength through the dewatering, the interlocking and densification of the system. However, under soaking conditions, it increases the swelling due to its water holding capacity and its massive growth, which can typically expand up to 140%, as reported by [46].

Conclusion

The efficacy of sulfate soil stabilisation by using a binary blend of cement and silica fume was investigated in this study through a physico-mechanical analysis, including unconfined compressive strength, linear expansion and derivative thermogravimetric analysis. The following conclusions can be shown as follows:

- The presence of sulfate in cemented soil experienced a massive expansion due to the formation and growth of ettringite; thus, the cementation of sulfate soil with cement is not recommended.
- Substitution of cement with silica fume yielded a gradual strength increase as the silica fume content increased to 30%, beyond which a counterproductive effect was observed.
- The swelling values of sulfate kaolin samples stabilised with binary blends of C–S are directly proportional to the cement amount and adversely proportional to silica fume amount, of which a blending proportion of 50% cement and 50% silica fume is the optimum for restricting the sulfate-induced expansion.
- The blending ratio of 7C-3S was optimal in terms of the strength performance, while the binary blend of 5C-5S was superior in terms of the expansion.

Overall, supplementation of cement with silica fume has been proven to be an effective solution for restricting the formation of ettringite in sulfate soil. However, further research study considering different sulfate types and different natural soils is needed to validate the optimal blends of cement and silica fume proportion.

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