

# **Influence of Supplementary Cementitious Materials on Sulfate Resistance of Mortar Mixtures**

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# *Abstract*

Sulfate reaction is a damaging expansive process that deteriorates cement-based structures over time. Various methods have been proposed to mitigate the effects of sulfate attack in concrete. Using Type II or Type V portland cement is an appealing approach to control sulfate attack. Additionally, incorporating supplementary cementitious materials (SCMs) such as class F fly ash, a byproduct of coal combustion for electricity generation, has proven effective at mitigating sulfate attack damage. However, future availability of fly ash is uncertain due to the energy industry transitioning towards more sustainable methods of energy production rather than relying on coal combustion. Consequently, there is an incentive to seek alternative SCMs that can effectively mitigate sulfate attack while being environmentally sustainable and economically feasible. In this study, ASTM C1012, a globally recognized standard test method for sulfate evaluation, was employed to assess sulfate resistance of mortar specimens. In total, 14 mortar mixtures containing various types and concentrations of alternative SCMs, including silica fume, metakaolin, and pumicite along with two types of portland cement (Type I and Type I/II) were produced. The results indicated that Type I/II portland cement had greater sulfate resistance compared to Type I cement in mortar mixtures, regardless of the type and concentration of SCMs used. Additionally, although metakaolin considerably improved sulfate resistance, silica fume and pumicite used in this study had only limited impact on sulfate resistance of the specimens. When evaluating ternary mixtures, using a combination of 22.5% metakaolin and 7.5% fly ash to replace 30% of Type I portland cement resulted in the greatest sulfate resistance among all mortar mixtures, with 0.054% expansion after nine months of testing. It is worth mentioning that when using Type I/II portland cement and only 15% metakaolin (as a cement replacement), sulfate resistance was comparable to the ternary mixture with 22.5% metakaolin, 7.5% fly ash, and with Type I portland cement. Overall, the results showed that metakaolin, fly ash, and pumicite can be considered effective SCMs for improving sulfate resistance*.* 

**Keywords:** *Fly Ash, Metakaolin, Mortar Mixtures, Pumicite, Silica Fume, Sulfate Resistance*

## **1. Introduction**

One of the important durability issues in concrete structures such as concrete pavement, bridge piers, dams, buried foundations, and piles is sulfate attack [1-4]. Researchers worldwide have extensively investigated the deteriorative effects of sulfate attack on reinforced concrete members caused by external sources such as groundwater [5-9]. External sulfate attack typically manifests in two forms: physical and chemical [10]. Physical attack results in surface cracking and concrete erosion caused by phase changes in sulfate solutions [11]. Chemical attack induces volumetric expansion in hardened concrete by altering hydration products within the cement paste, leading to cracking, increased permeability, and decreased strength [12-14]. This study primarily focuses on chemical sulfate attack, which includes complex phenomena that are not completely understood.

To prevent sulfate attack, it is often beneficial to add supplementary cementitious materials (SCMs) and use low water-to-cement (w/c) ratio. SCMs typically reduce permeability, thereby restricting penetration and movement of external sulfate ions within concrete that leads to an increase in sulfate resistance. Additionally, SCMs can reduce the availability of aluminate hydrates for reacting with external sulfate by consuming reactive tricalcium aluminate (C3A) in the cement paste [15]. Class F fly ash stands out as a highly effective supplementary cementitious material (SCM) for enhancing sulfate resistance in concrete. A byproduct derived from coal combustion in power plants, fly ash has a long history of use in improving not only the sulfate resistance of concrete but also its mechanical and durability properties. This dual benefit not only reduces material costs but also increases sustainability [16,17]. However, obtaining class F fly ash has become challenging for the concrete sector due to environmental considerations and shifts to the renewable energy sources in the energy sector. Therefore, it has become crucial to identify other cost effective and environmentally friendly SCMs that are capable of addressing sulfate attack effectively and are expected to be available for decades to come.

The main objective of this study was to examine how various SCMs affect the sulfate resistance of mortar mixtures. To accomplish this goal, 14 mortar mixtures utilizing two types of portland cement (Type I and Type I/II) along with different SCMs including fly ash, silica fume, pumicite, metakaolin, and combinations of these SCMs were produced. Sulfate attack testing was conducted following ASTM C1012 [18] to evaluate the resistance of the mortar mixtures against sulfate exposure.

## **2. Methodology**

#### *2.1 Materials*

The materials used in this study consisted of locally sourced sand and six types of cementitious materials. Sand was obtained from GCC in Las Cruces, NM, USA. Table 1 presents the gradation and physical properties for sand.

Two, It classified (percent passing) and proposed properties of same. Sieve Opening, mm							<b>Bulk Specific</b>	<b>Moisture</b>	Absorption,	<b>Fineness</b>
4.75	2.36	1.18	0.6	0.3	0.15	<b>PAN</b>	Gravity	Content, $\frac{0}{0}$	$\frac{0}{0}$	<b>Modulus</b>
95.2	82.7	71.2	53.3	18.3	1.53	0.0	2.54	0.41		2.78

Tab. 1. Gradation (percent passing) and physical properties of sand.

In this study, six cementitious materials were also used including a Type I portland cement produced by Texas Lehigh Cement (shipped from Buda, TX, USA), a Type I/II cement manufactured by GCC, Las Cruces, NM, USA, a class F fly ash (F) produced at the San Juan power plant in northern New Mexico, USA, a silica fume (S) produced by BASF Chemical Company, Cambridge, MA, USA, a pumicite (P) mined near Española, NM, USA, and a metakaolin (M) manufactured by Grace in Aiken, SC, USA. Chemical properties, chemical composition, and physical properties for the cementitious materials used in this study are presented in Table 2.



#### *2.2 Mixture Proportions*

A total of 14 mortar mixtures were produced in this study. Nine of the mixtures contained Type I and five of them contained Type I/II cement as the main cementitious material. Additionally, SCMs including fly ash (FA), silica fume (S), pumicite (P), metakaolin (M), or a combination of these SCMs were used in some of the mixtures to partially replace portland cement. The two control mortar mixtures (without SCM using only either Type I or Type I/II portland cements) were proportioned using ASTM C1012 [18] requiring mixtures without SCM to have a fixed w/c ratio of 0.485 and one part cement to 2.75 parts sand (by mass). For mixtures that also contained SCMs, ASTM C1012 [18] recommends that the water content should be adjusted to obtain a flow within the range of  $\pm 5\%$  compared to the control mixture. However, this adjustment generally leads to different water-to-cementitious materials (w/cm) ratios, causing more variability in the results of the sulfate attack tests since the w/cm ratio substantially impacts sulfate resistance [12]. Therefore, in this study, a fixed w/cm ratio of 0.485, the same as for the control mixtures, was used for mixtures containing SCMs. Table 3 presents the batch quantities for the mortar mixtures.



In the Mixture Name column in Table 3, the first letter and following number represent the type of cement (T1 for Type I portland cement and T2 for Type I/II portland cement) and the letters and following numbers after the hyphen show the type and concentration of the SCMs used. The SCM acronyms used in this study were F for fly ash, S for silica fume, P for pumicite, and M for metakaolin. The concentrations of each SCM is the ratio of the SCM to the total mass of cementitious materials (%). For instance, T1-M22.5F7.5 indicates a mixture with Type I portland cement, containing 22.5% metakaolin and 7.5% fly ash, both as percentages of the total cementitious materials by mass.

### *2.3 Mixing Procedures*

Mixing was performed according to ASTM C305 [19] in a 0.019 m3 capacity bucket mixer with an inclined axis of rotation. The components for each batch were mixed thoroughly before casting. Before any other ingredient, the water was introduced to the mixer. Subsequently, the cement was added to the water, and at a slow speed, mixer was activated. After 30 seconds, the sand was gradually added over the next 30 seconds while maintaining the slow mixing speed. Then, the mixer speed was increased to medium for an additional 30 seconds. Afterward, the mortar was allowed to rest for 90 seconds. During the initial 15 seconds of this pause, any mortar clinging to the sides of the bucket was promptly scraped down into the batch. For the remaining 75 seconds of this rest period, the mixing bucket was covered with a lid. Finally, the mortar underwent another 60 seconds of mixing at a medium speed. For each mixture, four bars measuring 25×25×285 mm, with gauge studs embedded in both ends, were cast.

#### *2.4 Testing procedures*

In this study, the ASTM C1012 [18] testing method was used to evaluate the sulfate resistance of mortar mixtures. Before demolding, specimens were held 25 mm above a water bath for the initial curing stage. The water temperature was maintained at 35±3℃. After 23.5±0.5 hours, the specimens were demolded and placed in a limewater tank at 23±2℃ for a three-day curing period. At an age of four days, initial length measurements were conducted, and the mortar bars were submerged in a container filled with a 5% sulfate solution (33,800 ppm SO42-). Each container held 3000 mL of the sulfate solution to provide a ratio of 4.0 for the solution volume to the specimen's volume as recommended by ASTM C1012 [18].

To track changes in expansion, length measurements were recorded at intervals of one, two, three, four, eight, 13, and 15 weeks, or more frequently if necessary. If the specimens did not fail after 15 weeks, or if a gradual and slight increase in expansion occurred during these specified intervals, additional measurements were taken at four, six, and nine months to ensure a thorough assessment of the mortar bars' long-term performance in the sulfate solution. The sulfate solution was replaced following each measurement interval to maintain a consistent sulfate exposure environment and to counteract leaching effects.

Following the completion of the ASTM C1012 [18] testing, the expansion results were analyzed using the criteria outlined in ACI 318-19 [20] to classify the different mortar specimens based on their sulfate resistance. Table 4 presents the maximum expansion limits specified by ACI 318-19 [20] for exposure classes S1, S2, and S3. The exposure severity increases from S1 to S3 and more stringent limitations on expansion are required to ensure adequate performance of cementitious materials combinations exposed to more severe environments.



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## **3. Results**

Table 5 provides a summary of the results obtained for all of the mortar mixtures. The six- and nine-month expansion results, as well as the average time required for specimens to reach the 0.05 and 0.1% expansions when tested according to ASTM C1012 [18], are presented because they are needed to classify a mixture into one of the sulfate exposure classes specified by ACI 318-19 [20].



#### *3.1 Cement Type Effects*

According to Table 5, mixtures without SCM (T1 and T2) were not suitable for any of the exposure classes specified by ACI 318-19 [20]. This is because they exceeded the maximum expansion limits for each of the sulfate exposure classes (0.1%) before six months of exposure to sulfate solution, indicating their inadequacy for sulfate-rich environments. However, a comparison between the results of mixtures T1 and T2 reveals that the Type I/II mixture (T2) took longer to reach the expansion limits compared to the Type I mixture (T1). This suggests that sulfate resistance of Type I/II cement was better than Type I cement, as expected. The increased sulfate resistance of Type I/II cement can primarily be attributed to its lower C3A content as shown in Table 2, resulting in reduced formation of expansive ettringite, and consequently, a decreased susceptibility to sulfate attack.

#### *3.2 Silica Fume Effects*

Replacing 7.5% of Type I cement with silica fume in mixture T1 to produce mixture T1-S7.5 did not improve sulfate resistance. Similar to mixture T1, mixture T1-S7.5 was not suitable for any of the exposure classes since the expansion of 0.1% was observed before the six-month mark, indicating that replacing 7.5% of portland cement with silica fume was not able to increase sulfate resistance in Type I mortar mixtures. The reason that silica fume failed to improve the sulfate resistance appears to be inadequate dispersion of densified silica fume particles within the mortar mixture, which is a common issue that has been identified by other researchers [21,22]. This insufficient dispersion may result from the absence of coarse aggregates in mortar mixtures. Typically, coarse aggregates aid in breaking up densified silica fume particles. Therefore, the condensed silica fume did not contribute significantly to improving the microstructure of the specimens through chemical reactions; rather, they primarily acted as filler. In some cases, such as mixture T1-S7.5, the inclusion of silica fume might even have a detrimental effect on the mortar mixture sulfate resistance. This negative impact could be ascribed to two main factors: firstly, the partial substitution of cement with a SCM that might not have dispersed adequately or undergone full reaction, and secondly, the ultra-fine silica fume particles could disrupt particle gradation, increasing permeability and facilitating the entry and movement of sulfate ions, thereby accelerating sulfate attack.

#### *3.3 Pumicite Effects*

Using pumicite as a cement mass replacement generally improved sulfate resistance of the mortar mixtures. This improvement can be attributed to the ability of pumicite to decrease permeability, improve pore structure, and the decreased calcium hydroxide (CH) and C3A contents (per unit volume) caused by using less portland cement. These combined effects likely contributed to the overall improvement against sulfate attack [23-27].

Within Type I mixtures, although increasing pumicite content from 0 to 7.5% or from 7.5 to 15% improved sulfate resistance, replacing either 7.5 or 15% of cement with pumicite did not lead to a change in exposure classes. Both T1-P7.5 and T1-P15 mixtures reached the 0.1% expansion limit before the six-month mark, indicating unsuitability for any sulfate exposure classes, similar to the mixture without pumicite (T1).

In the case of Type I/II mixtures, replacing 15% of Type I/II cement with pumicite (mixture T2-P15) significantly improved sulfate resistance, making it suitable for sulfate exposure class S1 according to ACI 318-19. Specimens from mixture T2-P15 reached the 0.05 and 0.1% expansion limits after 157 and 250 days, respectively. Since the expansion after 180 days was less than 0.1% (0.055%), this mixture could be considered suitable for sulfate exposure class S1 as specified by ACI 318-19 [20]. However, for this mixture to be considered suitable for exposure classes S2 and S3, the expansion at 12 months must be less than 0.1%. Ninemonth testing showed that specimens from this mixture reached 0.1% expansion prior to 12 months (at 250 days), indicating that this mixture cannot be accepted for sulfate exposure classes S2 and S3 as defined by ACI 318-19 [20].

Comparing Type I and Type I/II mortar mixtures with 15% pumicite (T1-P15 and T2-P15) indicates that the use of Type I/II portland cement significantly increased the testing time and transitioned the specimens from being unsuitable for any of the sulfate exposure classes (Type I mixture) to meeting the requirements for exposure class S1 (Type I/II mixture). This is primarily attributed to the lower  $C_3A$  content in the Type I/II cement compared to the Type I cement (Table 2) resulting in reduced formation of expansive ettringite.

## *3.4 Metakaolin Effects*

Generally, substituting a portion of the cement mass with metakaolin (7.5, 15, and 22.5%) resulted in enhanced sulfate resistance. Among the 14 mixtures examined in this research, only three were deemed suitable for all sulfate exposure classes (S1, S2, and S3) according to ACI 318-19 [20]. Interestingly, all of the promising mixtures contained either metakaolin alone or in combination with another SCM. The improved sulfate resistance achieved with metakaolin can be attributed to several factors. Firstly, its ultra-fine particles fill voids in the concrete, reducing the permeability of the microstructure that restricts sulfate ion penetration. Secondly, metakaolin reaction with CH results in the formation of secondary calcium silicate hydrate, which further densifies the pore structure and restricts sulfate ion movement. Additionally, the pozzolanic reaction between metakaolin and CH decreases the availability of CH for sulfate attack, thereby enhancing sulfate resistance [23-25].

In the case of Type I mixtures, although the addition of 7.5% metakaolin (T1-M7.5) led to considerable sulfate resistance improvements, this mixture (T1-M7.5) still did not meet the requirements of ACI 318-19 [20] for any of the exposure classes since 0.1% expansion occurred before six months. These findings are consistent with the results from T1-P7.5, where 7.5% of the cement mass was replaced with pumicite. This shows that replacing 7.5% of the Type I cement mass with SCMs, whether it is metakaolin or pumicite, is insufficient to achieve sulfate resistance levels that meet the requirements for any of the sulfate exposure classes. However, metakaolin showed a superior effect in improving sulfate resistance compared to pumicite.

Increasing metakaolin content from 7.5 to 15% resulted in the specimens transitioning from being unsatisfactory for any of the exposure classes to meeting the acceptance criteria for class S1. Further increase in the metakaolin content from 15 to 22.5% (T1-M22.5) ensured suitability for all sulfate exposure classes defined by ACI 318-19 [20].

Within the Type I/II mixtures, replacing 7.5% of the Type I/II portland cement with metakaolin significantly increased sulfate resistance compared to the mixture without metakaolin (T2). However, this improvement was insufficient to classify the specimens for any sulfate exposure classes, as they showed 0.1% expansion before six months. This finding was similar to the results obtained using Type I cement (T1-M7.5). Notably, when metakaolin content was increased from 7.5 to 15%, further improvement in sulfate resistance was observed, and specimens from this mixture (T2-M15) became suitable for all sulfate exposure classes specified by ACI 318-19.

Figure 1 illustrates the expansion versus time for mixture T2-M15. This graph reveals that expansion increased at a moderate rate until reaching 0.025% expansion (at approximately 230 days), followed by a significant increase in slope, indicating the initiation of cracks in the specimens, subsequent ingress of the sulfate solution, and the expansive sulfate attack reactions. This pattern of a slight plateau followed by an abrupt slope increase due to cracking was consistent with behaviors of other specimens.

#### *3.5 SCMs Combination Effects*

Specimens from the mixtures T1-M7.5S7.5 and T2-M7.5S7.5, containing 7.5% metakaolin and 7.5% silica fume, failed to meet the criteria set forth by ACI 318-19 [20] for any sulfate exposure class since they showed 0.1% expansion in less than six months.

Another ternary mixture (T1-M22.5F7.5) containing 22.5% metakaolin and 7.5% fly ash showed expansions of 0.023 and 0.054% after 180 and 270 days of testing, respectively. Since the expansion was less than 0.1% after six months (0.023%), the mixture T1- M22.5F7.5 met the requirements for exposure class S1 according to ACI 318-19 [20]. These specimens were deemed suitable for exposure classes S2 and S3 as well, which are more severe than S1. This is because the expansion at six months was less than 0.05%. These results show that the mixture T1-M22.5F7.5 outperformed other mortar mixtures tested in terms of sulfate resistance (least expansion) and was one of only two Type 1 mortar mixtures, along with T1-M22.5, that met the requirements for all exposure classes. This remarkable performance can be attributed to several factors: 1) including fly ash in the mixture, 2) the synergistic effects of the metakaolin and fly ash, and 3) the overall greater content of SCM (30%) in the mixture. These factors collectively contributed to reducing permeability, enhancing pore structure, and lowering CH and C3A contents (per unit volume).

Comparing mixture T1-M22.5F7.5 and mixture T1-M22.5 indicates that the inclusion of additional 7.5% fly ash in mixtures already containing 22.5% metakaolin significantly prolonged the testing time (improved sulfate resistance). The specimens with fly ash (T1- M22.5F7.5) showed approximately half the expansion observed in mixtures without fly ash (T1-M22.5) at the same testing age (0.037% compared to 0.023% for a 180-day testing period and around 0.1% compared to 0.054% for a 270-day testing period).



Fig. 1. Expansion results for the mixture T1-M22.5F7.5.

## *3.6 Variation of Results*

Table 6 presents the standard deviation (SD) and coefficient of variation (CoV) values for the testing time results obtained in this study. Four bars were tested for each mortar mixture. It should be noted that, in the case of all pumicite mixtures (T1-P7.5, T1-P15, and T2-P15), one specimen was fractured immediately after demolding, leaving only three specimens available for assessment.



Comparing SD and CoV values for testing time between the 0.05% and 0.1% expansion limits shows that Type I/II mixtures generally exhibited lower SD and CoV values compared to Type I mixtures. This trend was anticipated since Type I specimens are expected to undergo more cracking, exposing additional readily available C3A. However, deviations from this trend were noted in mixtures containing 7.5% metakaolin and 7.5% silica fume, which showed higher SD and CoV values for the Type I/II mixture.

The range of SD and CoV values for testing time was found to be comparable for both expansion limits. For the 0.05% expansion limit, SD values ranged from 0.64 to 39.2 days, and for the 0.1% expansion limit, SD values ranged from 0.72 to 37.6 days. Similarly, CoV values ranged from 1.25 to 56.7% for the 0.05% expansion limit and from 1.60 to 49.6% for the 0.1% expansion limit. Pumicite mixtures generally showed higher SD and CoV values compared to other mixtures, with mixtures containing 15% pumicite and Type I cement (T1-P15) showing the highest SD value of 39.2 days and CoV value of 56.7% among all tested mixtures for both 0.05 and 0.1% expansion limits. This higher SD and CoV values in pumicite mixtures can be attributed to the less homogenous nature of pumicite compared to manufactured SCMs such as metakaolin or silica fume.

## **4. Conclusions**

Based on the conducted research, the following conclusions were drawn:

- 1. Mortar mixtures without SCM, regardless of whether Type I or Type I/II portland cement was used, were not suitable for sulfate exposure.
- 2. Type I/II cement improved sulfate resistance compared to Type I cement, regardless of the SCM type and concentration. This outcome was anticipated due to the lower C3A content present in Type I/II cement.
- 3. Replacing 7.5% of cement with silica fume in binary and ternary mortar mixtures did not improve sulfate resistance, possibly due to the inadequate dispersion and consequent limited reactivity of condensed silica fume particles caused by the lack of coarse aggregates in the mortar mixtures.
- 4. Replacing cement with metakaolin (7.5, 15, or 22.5%) or pumicite (7.5 or 15%) in mortar mixtures improved sulfate resistance. This enhancement seems to stem from the inherent capability of SCMs to reduce permeability and enhance pore structure, alongside the decreased presence of CH and C3A resulting from the reduced use of portland cement.
- 5. The ternary mixture containing 22.5% metakaolin and 7.5% fly ash outperformed all other mixtures and showed the least expansion among all specimens.

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