EFFECT OF SULFATE AND CHLORIDE SOLUTIONS ON THE COMPRESSIVE AND FLEXURAL STRENGTHS OF MORTAR INCORPORATING SPENT COFFEE GROUNDS

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Abstract - Vietnam is a major coffee consumer, generating large amounts of spent coffee grounds (SCG), typically discarded as waste. This creates environmental concerns but also offers potential for resource reuse. Meanwhile, Vietnam's construction industry faces a shortage of natural river sand. The study explores the potential of using SCG as a partial replacement for natural sand in mortar production, aiming to support sustainable development. The effect of varying SCG contents on the compressive and flexural strengths of mortar under different aggressive environments was examined, including immersion in water, 5% NaCl solution, 5% Na_2SO_4 solution, and a mixed 5% NaCl-Na_2SO_4 solution. Results revealed that both compressive and flexural strengths increased over time; however, higher SCG content resulted in a gradual decrease in both. Furthermore, immersion in different solutions influenced the mortar's strength, with tap water proving the most beneficial for compressive strength, while 5% Na_2SO_4 solution showed the greatest effect on flexural strength.

Key words - Mortar; Spent coffee grounds; Sulfate; Chloride; Compressive strength

1. Introduction

SCG is a solid waste by-product directly linked to coffee consumption. With an estimated 2 billion cups of coffee consumed worldwide each day [1], the resulting volume of SCG waste is substantial. Currently, the majority of SCG is disposed of in landfills, where they undergo decomposition. During this process, SCG contributes to soil humus formation but simultaneously releases methane (CH_4) into the atmosphere. Methane is a potent greenhouse gas with a global warming potential 21 times greater than that of carbon dioxide (CO_2) [2]. Additionally, the decomposition of SCG emits other greenhouse gases such as carbon dioxide, nitrous oxide, and ammonia [3]. Therefore, there is an urgent need to explore sustainable recycling solutions to convert this type of waste into value-added products.

Several studies worldwide have investigated the utilization of SCG in the construction sector. SCG can be blended with various binders and other waste materials to produce road subbase materials [4]. They have also been employed in brick manufacturing as an additive, increasing water absorption, reducing thermal conductivity, and compressive strength [5]. At a ratio of 1-2% SCG by weight relative to clay, the bricks exhibit lower compressive strength and thermal insulation compared to the control clay bricks without SCG. However, when the SCG content is increased to 3-5%, the bricks have lower density and higher porosity, significantly improving thermal insulation while maintaining adequate mechanical strength. The optimal SCG content is 3%, at which the compressive strength of the brick exceeds that of the conventional clay brick [6]. Additionally, SCG has been explored as a sound-absorbing material [7], lightweight ceramic aggregates [8], and as a gypsum-based material to reduce thermal conductivity and diffusivity [9]. SCG has also been studied for its potential in sustainable construction materials, such as substituting SCG for Portland cement [10].

Several studies have found that the thermal treatment of SCG can yield positive results for construction applications [11–12]. Depending on the specific thermal processing conditions, either activated carbon or biochar can be produced. Activated carbon and biochar share many similar properties, making it difficult to clearly distinguish one from the other. Na et al. [11] investigated the effect of activated carbon from SCG on the mechanical properties of cement mortar at various replacement levels (0%, 1%, 1.5%, 5%, and 10% by weight of cement). SCG was physically activated at 600°C for 1 hour. Results showed that adding up to 1.5% (by cement weight) improved compressive strength, while contents above 5% significantly reduced it. Roychand et al. (2023) reported that untreated SCG released organic compounds that hindered cement hydration, significantly reducing the compressive strength of concrete. In contrast, pyrolyzing SCG decomposed the organic matter, producing carbon-rich, porous biochar. Higher pyrolysis temperatures increased biochar porosity. Notably, SCG pyrolyzed at 350°C (350CBC) improved concrete performance, with a 29.3% increase in compressive strength at a 15% sand replacement level. It is evident that thermal treatment, as demonstrated in studies [11-12], helps stabilize the material properties of SCG. However, the high energy consumption involved limits its practical application. Recently, Lee et al. [13] examined the use of raw dried SCG in mortar without thermal treatment, with SCG contents of 0.6%, 0.9%, 1.8%, 3.0%, and 6.0% by weight of cement, corresponding to 1%, 1.5%, 3%, 5%, and 10% sand volume replacement. Mortars with 0.6% and 0.9% SCG showed different trends from the control, with compressive strength decreasing by 24.5% and increasing by 8.3%, respectively. For SCG contents above 3.0%, high water absorption of SCG led to strength loss due to incomplete hydration.

Coffee is the second most traded commodity in the world after crude oil, and Vietnam is among the largest coffee-producing countries, ranking just behind Brazil. As a result, the quantity of SCG produced in Vietnam is considerable. Therefore, identifying recycling solutions to transform this waste into a valuable resource is imperative. The construction industry holds significant potential for increasing the recycling rate of this waste material, contributing to sustainable development from an environmental perspective. Furthermore, rapid urbanization, infrastructure expansion, and housing development have intensified the demand for natural construction materials, such as river sand, which are becoming increasingly scarce. Compressive strength and flexural strength are among the most critical properties to consider when evaluating the load-bearing capacity of mortar. A sustainable material must be capable of withstanding chemical, physical, or biological degradation throughout the service life of a structure. Sulfate attack is a common form of deterioration in concrete structures [14]. Due to the widespread presence of sulfate-containing soils and saline water, sulfate-induced degradation has led to significant economic losses in infrastructure [15-16]. Consequently, extensive research has been conducted to understand the mechanisms of sulfate attack in concrete and the various factors influencing this process [17-19]. In addition to sulfates, chloride ions can also penetrate concrete and mortar, transported from chloride-containing environments, either as impurities in the materials used or during the construction process. In coastal regions, structures located near the sea, where groundwater is rich in chloride and sulfate ions, are particularly vulnerable to such chemical attacks [20].

To date, no studies have been conducted on the behavior of mortar incorporating SCG under the influence of corrosive environments. Most previous studies typically used thermally treated SCG, whereas this study focuses on the direct application of untreated SCG. Moreover, the impact of SCG on the mechanical strength of construction materials remains inconclusive. Therefore, this study investigates the use of SCG as a partial replacement for sand in mortar production and evaluates the compressive strength and flexural strength of the SCG-modified mortar when exposed to different aggressive environments, including water, 5% NaCl solution, 5% Na_2SO_4 solution, and a mixed solution of 5% NaCl and 5% Na_2SO_4. The results of this study will reveal the load-bearing capacity of mortar under different aggressive environments, serving as important data for evaluating the potential of replacing sand with SCG. Further experimental investigations are needed to gain deeper insights into the properties of mortar under aggressive environments, such as chloride ion permeability and the formation of corrosion products like ettringite. Some related properties, including water absorption, drying shrinkage, and sulfate expansion of mortar containing SCG, are discussed in the study by Nguyen et al. [21].

2. Materials and Experimental Programs

2.1. Materials

The materials used for preparing the mortar specimens in this study include: PCB40 cement, lime, natural sand, SCG, water, and superplasticizer (SP). Specifically, PCB40 cement manufactured by Bim Son Cement Plant was used, with a specific gravity of 3.12 t/m^3. The main chemical composition of the cement is presented in Table 1. Cement primarily consists of SiO_2 and CaO, with a combined content reaching 77.75% by mass. Hydrated lime with a specific gravity of 2.21 t/m^3 was added in a small amount (1% of the total binder content) to the mortar samples containing SCG to enhance their setting acceleration. In this study, the binder (abbreviated as B) is a mixture of cement and the added lime. The natural sand, commercially available, had a specific gravity of 2.62 t/m^3, a bulk density (dry, natural state) of 1.45 t/m^3, a moisture content of 0.53%, and a water absorption capacity of 0.42%. Before use, the sand was sieved to remove particles retained on a 1.25 mm sieve and those passing through a 0.14 mm sieve. Table 2 presents the sieve analysis and fineness modulus of the natural sand, which was determined to be 1.21. SCG was collected from local coffee shops in Thanh Hoa City, then washed to remove impurities and dried. According to Ballesteros et al. [22], SCG is composed of cellulose (12.4 ±0.79%), hemicellulose (39.1 ±. 1.94%), lignin (23.9 ±1.70%), lipids (2.29 ±0.3%), proteins (17.44 ±0.1%), and total dietary fiber (60.46 ±2.19%). In addition, SCG contains various micronutrients, including potassium, calcium, magnesium, sulfur, phosphorus, iron, manganese, boron, and copper. The SCG in this study has a specific density of approximately 900 kg/m^3, indicating that it has high porosity, similar to the results from previous studies [13]. Moreover, microstructural analyses of SCG, as reported in studies [6, 10, 13], reveal a highly porous structure characterized by numerous voids and a rough surface texture. Before use, the SCG was washed thoroughly, soaked in a 2% NaOH solution for 2 hours, then boiled at 90-100^{o}C for 2 hours. This treatment aimed to remove impurities such as sugar, milk residues, and other contaminants from the SCG. Afterward, the SCG was placed on a 0.14 mm mesh sieve to allow excess water to drain out. As a result, the SCG used in this study was in a water-saturated condition. This detail is important because SCG has high porosity. If used in a dry state, it would require a significant amount of additional water due to its high water absorption capacity. The use of materials in a saturated water condition before mixing has been used for highly porous materials, such as waste incineration bottom ash, as studied by Huynh and Ngo [23]. The particle size of SCG has been reported to range from 0.225mm to 0.550mm [10]. Figure 1 presents the image of SCG after undergoing the preliminary treatment steps as described above. A type F superplasticizer (SP), mainly composed of Naphthalene Formaldehyde Sulfonate and with a specific gravity of 1.15 t/m^3, was used to adjust the workability of the mortar, aiming to achieve a spread diameter of 16 ± 2 cm.

Table 1. *Main chemical compositions of cement (%)*

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	LOI*
Cement	22.30	6.68	4.73	55.45	2.40	1.28	0.74	0.45

LOI= Loss of ignition*

Table 2. *The sieve analysis and modulus of natural sand*

Sieve size (mm)	1.25	0.63	0.315	0.14	FM*
Cumulative percent retained (%)	0	5.1	16.0	100	1.21

FM=Fineness modulus*

Figure 1. *Spent coffee grounds*

2.2. Mix design and proportions

The mortar samples in this study were designed using the absolute volume method, with detailed mix compositions provided in Table 2. Lime was incorporated at a dosage of 1.0% by binder mass, partially replacing cement. The experimental process revealed that mortar samples containing SCG exhibited delayed setting, and lime powder was added to improve this property. The mortar mixtures were prepared with a constant aggregate-to-binder (A/B) ratio of 2.0. The aggregate component (denoted as A) consisted of natural sand and SCG. The content of SCG, denoted as (SCG/B), was 0%, 5%, 10%, 15%, and 20% by mass relative to the binder content (cement + lime). The mixtures were labeled as SCG*x*, where "*x*" denotes the percentage of SCG by weight relative to the total binder content. A fixed water-to-binder (W/B) ratio of 0.4 was maintained across all mixtures. The superplasticizer dosage was adjusted to ensure that the flow diameter of each mortar mix remained within 16 ± 2 cm, in accordance with the Vietnamese standard TCVN 4314:2022 [24]. The objective of this mortar mix design was to evaluate the influence of SCG incorporation on the CS and FS of mortar under normal (water) and corrosive environmental conditions.

2.3. Sample preparation and test method

Based on the compositions outlined in Table 3, all materials, including cement, lime, sand, and SCG, were accurately measured and prepared prior to mixing in the laboratory. Figure 2 illustrates the sieved and dried materials before the mixing process. The dry mix, consisting of fine aggregates (natural sand and SCG) and binders (cement and lime), was first added to the mixing pan and thoroughly blended for two minutes. Afterward, water combined with SP, as specified in Table 3, was gradually introduced into the mixture until a homogeneous consistency was obtained.

Table 3. *Mix proportions*

Mixture	Ingredient proportions (kg/m^3)					
	Cement	Lime	Sand	SCG	Water	SP
SCG00	671.4	0.0	1342.7	0.0	268.5	6.7
SCG05	648.2	6.5	1276.7	32.7	261.9	6.5
SCG10	633.1	6.4	1215.0	63.9	255.8	6.4
SCG15	618.6	6.2	1156.0	93.7	250.0	6.2
SCG20	604.9	6.1	1099.7	122.2	244.4	6.1

Figure 2. *Material preparation for one mixing batch*

A flow test was conducted in accordance with TCVN 3121-3:2003 [25] to ensure that all samples achieved a flow diameter within the range of 16 ± 2 cm by adjusting the amount of SP. Once the desired flow consistency was achieved, the mortar mixture was poured into steel molds with dimensions of 4 × 4 × 16 cm to form specimens for flexural and compressive strength testing. The mortar samples were demolded one day after casting and then immersed in the designated curing solutions until the experiments were conducted. Figure 3 shows the mortar samples after demolding. Figure 4 illustrates the mortar samples submerged in the respective solutions, including a 5% NaCl solution, a 5% Na_2SO_4 solution, and a mixed solution containing 5% NaCl and 5% Na_2SO_4. Tap water was used as a control for comparison. All solutions were refreshed every 28 days to maintain consistent chemical concentrations until the tests were performed. The purpose of this immersion was to evaluate the mechanical performance of the mortar samples under different environments.

Figure 3. *Mortar samples after demolding*

The flexural and compressive strength tests were performed in accordance with the procedures outlined in TCVN 3121-11:2003 [25]. Compressive strength was determined at 3, 7, 14, 28, and 56 days, while flexural strength was assessed at 14, 28, and 56 days. The reported results represent the average value from at least three specimens. For compressive strength, the average value was calculated from six specimens, as the prisms tested for flexural strength (4 × 4 × 16 cm) were broken in half. Each half-prism was then used for compressive strength testing on a compression apparatus with a cross-sectional area of 4 × 4 cm. As three specimens were tested for flexural strength, six additional halves were used for compressive strength testing.

***Figure 4.** Mortar samples immersed in different solutions*

3. Results and discussion

Compressive strength and flexural strength are critical parameters for evaluating the mechanical performance of mortar specimens. In this section, the compressive and flexural strengths are examined with respect to the influence of SCG as a partial sand replacement, as well as the effects of various aggressive environmental conditions.

3.1. Compressive strength

The compressive strength of mortar specimens containing SCG as a sand replacement was determined at 3, 7, 14, 28, and 56 days of curing. Figures 5, 6, 7, and 8 illustrate the development of compressive strength of mortar samples with varying SCG content, which were immersed in tap water, 5% NaCl solution, 5% Na_2SO_4 solution, and a mixed solution containing 5% NaCl and 5% Na_2SO_4, respectively. Based on the data presented in Figures 5 through 8, Figures 9 and 10 illustrate the effect of SCG content on the compressive strength of mortar samples immersed in different solutions at 28 and 56 days of curing, respectively.

The experimental results reveal a general trend of increasing compressive strength with curing age. For example, the compressive strength of the mortar sample containing 5% SCG increased from 25.8% to 100.8% in water, from 19.2% to 98.9% in the 5% NaCl solution, from 23.8% to 130.5% in the 5% Na_2SO_4 solution, and from 30.2% to 132.9% in the mixed solution of 5% NaCl and 5% Na_2SO_4, over the period from 7 to 56 days. The development of compressive strength over time can be primarily attributed to the continued hydration of cement, which enhances the microstructure and densifies the matrix as curing progresses.

Figures 5, 6, 7, and 8 clearly demonstrate a trend of decreasing compressive strength with increasing SCG content, especially when the sand replacement ratio reaches 15% and 20%. This reduction is attributed to the porous and highly absorptive nature of SCG, which results in a less compact microstructure as the SCG content in the mix increases. Consequently, the compressive strength of the mortar significantly decreases with higher SCG content. Although the compressive strength of the SCG-incorporated samples is considerably lower than that of the control mix (SCG00), at 28 days, the sample with the highest SCG replacement (SCG20) still achieved compressive strengths of 17.4 MPa, 12.41 MPa, 19.02 MPa, and 12.73 MPa when cured in tap water, 5% NaCl solution, 5% Na_2SO_4 solution, and the mixed NaCl and Na_2SO_4 solution, respectively, corresponding to mortar grades in the M10 to M15 range [24].

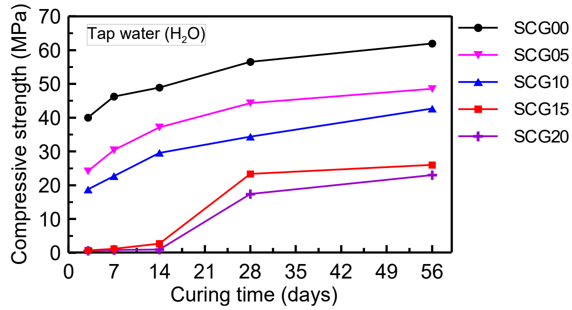

***Figure 5.** Compressive strength of mortars immersed in tap water*

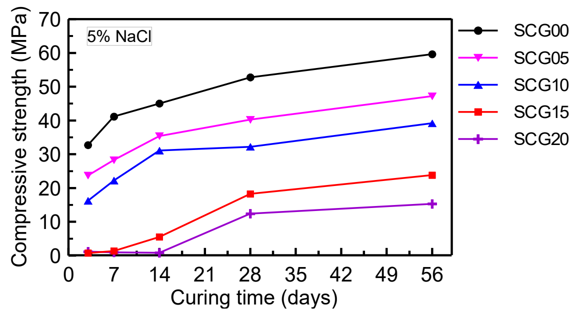

***Figure 6.** Compressive strength of mortars immersed in a 5% NaCl solution*

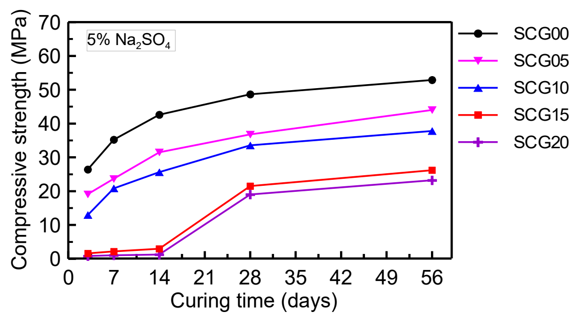

***Figure 7.** Compressive strength of mortars immersed in a 5% Na_2SO_4 solution*

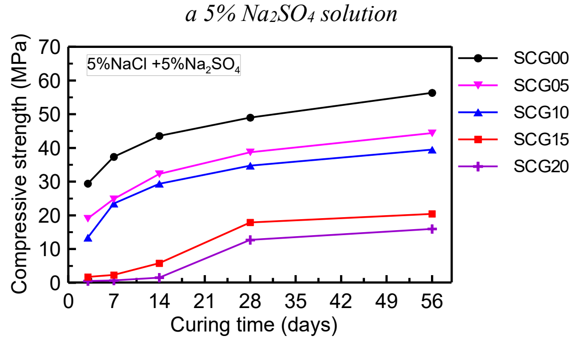

***Figure 8.** Compressive strength of mortars immersed in a 5% NaCl and 5% Na_2SO_4 solution*

An interesting observation in Figures 5, 6, 7, and 8 is that the samples containing 15% to 20% SCG exhibit a distinct strength development pattern, with a slow increase in compressive strength during the first 14 days, followed by a significant strength development after 14 days.

As illustrated in Figures 9 and 10, mortar samples immersed in tap water exhibited higher compressive strength compared to those exposed to a 5% NaCl solution. This finding appears to contradict established research by Camacho et al. [26] and Du et al. [27-28], who have demonstrated that variations in chloride concentration exert minimal influence on the compressive strength of concrete relative to specimens cured in tap water. The collective evidence from these investigations suggests that prolonged exposure to NaCl solutions does not significantly compromise the compressive strength of concrete. For instance, Du et al. [27] reported fluctuations in compressive strength across different immersion durations, with no consistent trend favoring either curing condition. Moreover, Du et al. [28] observed a marginal increase in compressive strength in concrete incorporating NaCl compared to the control mix. When NaCl is introduced into a mortar, part of it chemically interacts with the cement hydration products to form stable Friedel's salt [28]. The remaining chloride ions are present as free Cl^- in the pore solution. Notably, the formation of Friedel's salt is a volumetrically stable process that neither generates expansive forces nor detrimentally affects the microstructure or mechanical properties of the mortar [26-28].

From Figure 9 and most notably in Figure 10 (at 56 days of curing), it can be observed that mortar samples containing SCG contents ranging from 0% to 10%, when immersed in Na_2SO_4 solution, exhibit the lowest compressive strength compared to those immersed in other solutions. This result is consistent with the findings of experimental studies by Du et al. [27-28], which compared the compressive strength of concrete immersed in Na_2SO_4 solution to that of concrete immersed in solutions containing both NaCl and Na_2SO_4. However, for the SCG15 and SCG20 mortar samples, the compressive strength observed in Na_2SO_4 solution was equivalent to that in water and higher than in the other solutions. This may be due to the fact that at higher SCG content, the mortar samples, which contain a higher number of pores, are more susceptible to attack by Na_2SO_4, leading to the formation of gypsum, followed by the generation of ettringite through the reaction of gypsum with the cement hydration products [28-29]. As one of the corrosion products induced by sulfate ions, the amount of gypsum increases as the aluminum content in the cement is consumed. Both gypsum and ettringite are low-solubility salts, and their formation significantly increases the solid phase volume in the mortar, filling the pores and enhancing the strength of the mortar.

Finally, when the mortar is immersed in a mixed NaCl and Na_2SO_4 solution, the compressive strength of the mortar fluctuates within a range between that of the mortar immersed separately in NaCl and Na_2SO_4 solutions. This has been observed in numerous experiments, and researchers have consistently concluded that the presence of chloride can inhibit sulfate attack on concrete. The primary mechanism is thought to be the higher diffusion rate of Cl^- ions compared to SO_4^{2-} ions in concrete. Additionally, the presence of Cl^- ions may lead to the formation of Friedel's salt, thereby reducing the formation of sulfate corrosion products such as ettringite and gypsum [27-31].

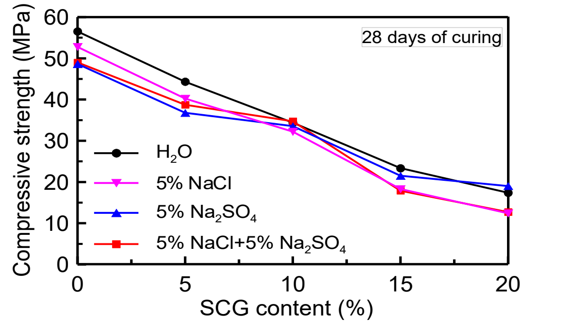

Figure 9. *Compressive strength of mortars at 28 days of curing*

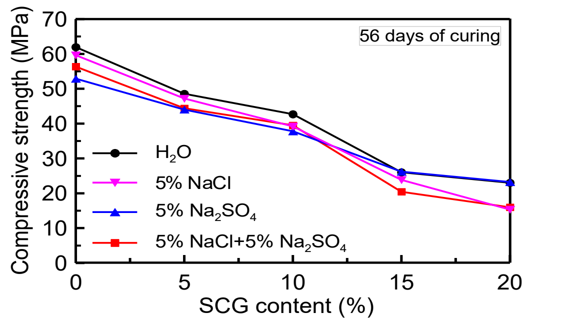

Figure 10. *Compressive strength of mortars at 56 days of curing*

3.2. Flexural strength

The flexural strength of mortar specimens incorporating SCG as a sand replacement was evaluated at curing ages of 14, 28, and 56 days. Figures 11 to 14 depict the progression of flexural strength in mortar samples with varying SCG contents, each immersed in different solutions: tap water, a 5% NaCl solution, a 5% Na_2SO_4 solution, and a mixed solution containing both 5% NaCl and 5% Na_2SO_4. To further illustrate the influence of solutions, Figures 15 and 16 present the flexural strength results of the mortar samples at 28 and 56 days of curing, respectively, for each immersion solution.

Figures 11 to 14 illustrate that the flexural strength of the mortar increases over time, following a trend similar to that of the compressive strength. This improvement is attributed to the cement hydration process, which gradually enhances the mortar's microstructure, making it denser and more resistant to tensile stresses. Furthermore, it is observed that as the SCG content increases, the flexural strength of the mortar decreases. This reduction is due to the porous and highly absorbent nature of SCG, which leads to a less dense microstructure as the SCG content in the mixture rises.

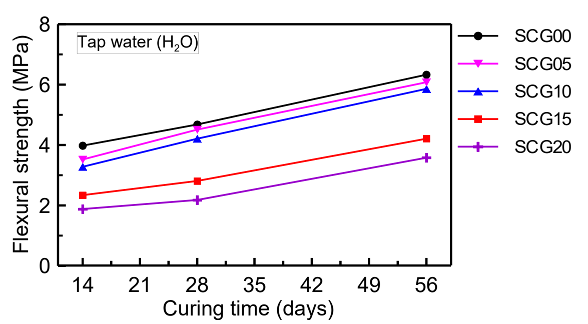

Figure 11. *Flexural strength of mortars immersed in tap water*

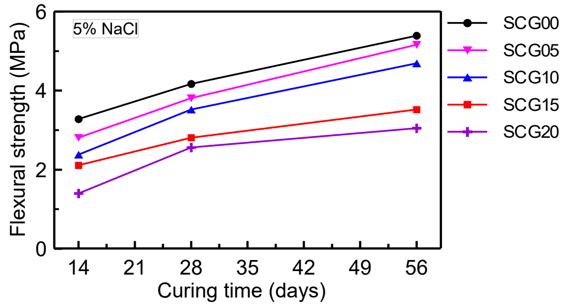

Figure 12. *Flexural strength of mortars immersed in a 5% NaCl solution*

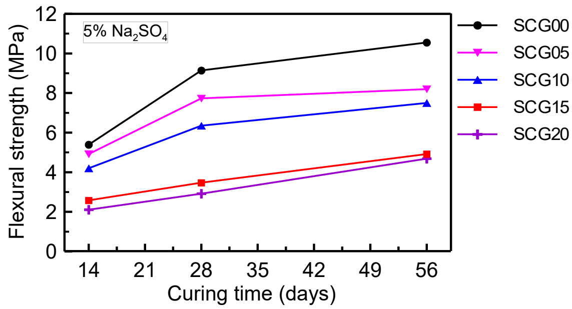

Figure 13. *Flexural strength of mortars immersed in a 5% Na_2SO_4 solution*

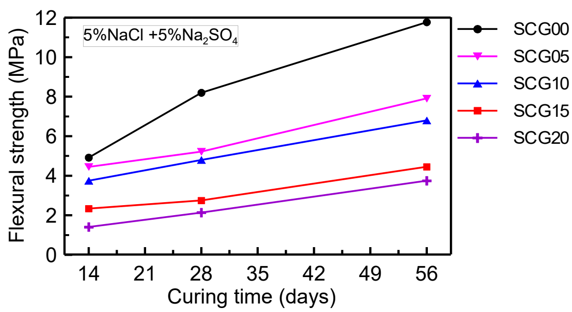

Figure 14. *Flexural strength of mortars immersed in a 5% NaCl and 5% Na_2SO_4 solution*

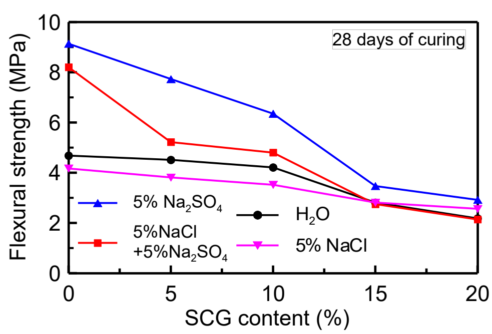

Figure 15. *Flexural strength of mortars at 28 days of curing*

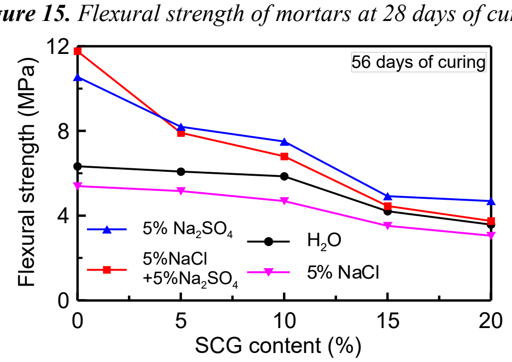

Figure 16. *Flexural strength of mortars at 56 days of curing*

Figures 15 and 16 show that the flexural strength of the mortar at 28 and 56 days of age exhibits a certain degree of variability. However, it is evident that the flexural strength of the samples immersed in water is higher than that of the samples immersed in NaCl solution, which is consistent with the observations made for compressive strength. Interestingly, the flexural strength of the mortar samples immersed in Na_2SO_4 solution is the highest, followed by those immersed in the mixed NaCl and Na_2SO_4 solution. This can be explained by the fact that flexural strength is determined through a three-point bending test, in which the load is applied at the midpoint of a simply supported beam. Failure in this setup occurs due to tensile stress at the bottom of the specimen. In samples immersed in sulfate solution, the formation of ettringite and gypsum leads to expansion, causing the mortar matrix to be subjected to triaxial compressive stresses. When tensile stress is applied through bending, these existing compressive stresses may contribute to an increase in tensile (flexural) strength. Further studies are needed to provide additional evaluation with a wider range of SCG contents and longer immersion durations.

4. Conclusion

This study was conducted to evaluate the impact of using SCG as a partial replacement for natural sand in the pursuit of sustainable development. In this study, SCG contents of 5%, 10%, 15%, and 20% by mass were used, based on the total binder content consisting of cement and lime. The study assesses the effect of SCG content on the compressive strength and flexural strength of mortar in various aggressive environments by immersing samples in water, 5% NaCl solution, 5% Na_2SO_4 solution, and a mixed 5% NaCl and 5% Na_2SO_4 solution. Several key conclusions can be drawn as follows:

- Both the compressive strength and flexural strength of the mortar samples increase over time. For a given solution and at the same age of curing, as the SCG content increases, both the compressive strength and flexural strength gradually decrease.

- The order of the beneficial effects of the immersion solutions on the compressive strength of mortar samples in this study is as follows: Tap water > 5% NaCl solution > 5% NaCl + 5% Na_2SO_4 solution > 5% Na_2SO_4 solution.

- The influence of the immersion solutions on the flexural strength in this study follows this order of effectiveness: 5% Na_2SO_4 solution > 5% NaCl + 5% Na_2SO_4 solution > Tap water > 5% NaCl solution.

As natural sand resources become increasingly scarce, coffee grounds may serve as a potential alternative for partially replacing natural sand. However, mortar containing SCG, particularly when used in aggressive environments exposed to chloride or sulfate ion attack, must be carefully and thoroughly evaluated. The proportion of SCG should be determined based on the load-bearing requirements of each structural element to ensure compliance with established technical standards. Future studies should consider a broader range of water-to-binder (W/B) and aggregate-to-binder (A/B) ratios, as well as evaluate different treatment methods for SCG, in order to more comprehensively assess the development of the technical properties of mortar samples incorporating SCG.

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