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Scientific paper

ISSN 0351-9465, E-ISSN 2466-2585

<https://doi.org/10.62638/ZasMat1313>



Zastita Materijala 66 (4)
820 - 828 (2025)

Influence of lime sludge addition on the mechanical and microstructural performance of pond ash based geopolymer concrete

ABSTRACT

This study investigates the addition of varying lime sludge (LS) proportions, and curing durations (3, 7, and 28 days) on the properties of geopolymer concrete (GC). All GC mixes were prepared with an 8 M sodium hydroxide solution and a fixed liquid-to-binder ratio of 0.4. The mechanical properties of the LS-based GC mixes were assessed and than control mix comprised entirely of pond ash (100% PA). Additionally, scanning electron microscopy (SEM) analysis was performed to investigate the microstructural differences between the control mix (100% PA, LSGC0) and the optimized mix with 50% PA and 50% LS (LSGC5). The results revealed that the LSGC5 mix achieved notable improvements in compressive, split tensile, and flexural strengths compared to the LSGC0 mix. SEM analysis highlighted the formation of a denser and more cohesive microstructure in the LSGC5 mix, attributed to the enhanced generation of calcium-alumino-silicate-hydrate (C-A-S-H) gels, which were less prevalent in the 100% PA mix. These findings demonstrate the efficacy of lime sludge as a sustainable replacement material, significantly enhancing the mechanical and microstructural properties of GC while reducing dependency on traditional cementitious components.

Keywords: Geopolymer concrete; pond ash; lime sludge; mechanical properties; microstructural properties.

1. INTRODUCTION

Concrete has long been recognized as a cornerstone material in modern construction, valued for its exceptional strength, versatility, and durability. Its significance is evident in its widespread use across a myriad of applications, including infrastructure projects such as roads, bridges, and dams, as well as residential and commercial buildings [1]. Ordinary Portland Cement (OPC) serves as the cornerstone of conventional concrete, widely recognized for its role as a binding agent and its contribution to the material's global prevalence. However, the production of OPC poses substantial environmental challenges despite its widespread utility [2,3]. The process involves the decarbonization of limestone and the intensive use of fossil fuels, resulting in substantial CO₂ emissions.

Moreover, with the accelerating pace of industrialization, the depletion of natural resources required for OPC production has reached alarming levels. These factors, combined with the high energy demands of cement manufacturing, underscore the urgent need for sustainable alternatives [4]. One promising innovation addressing these challenges is geopolymer concrete (GC), an eco-friendly substitution to cement. Geopolymers are synthesized from industrial by-products, which react with solutions to form a robust and durable binding matrix. This technology not only minimizes reliance on non-renewable resources but also repurposes waste materials, significantly reducing the carbon footprint of construction [5]. In addition to being environmentally sustainable, GC demonstrates superior resistance to chemical and thermal degradation, offering enhanced durability in harsh environments. By integrating geopolymer technology, the construction industry can mitigate its environmental impact while meeting the growing demand for sustainable infrastructure solutions [6].

The term "Geopolymer (GP)", introduced by Davidovits, describes a class of materials derived from the chemical synthesis of industrial by-products and agricultural residues. These

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Paper received: 08.12.2024.

Paper corrected: 24.01.2025.

Paper accepted: 28.01.2025.

materials, either used individually or in combination, include coal ash, slag, rice husk ash, oil palm ash, and bagasse ash [7]. Such residues serve as the primary source materials for the production of GP concrete, offering an innovative and sustainable approach to construction [8]. A critical component of the geopolymerization process is the alkaline solution, typically comprising potassium- or sodium-based soluble compounds. When these alkaline solutions activate the alumino (Al) -silicate (Si) content in the source materials, a series of chemical response occur, culminating in the formation of a geopolymer resin. The polymerization process initiates with the rapid decomposition of reactive Al-Si structures, releasing free SiO_4 and AlO_4 tetrahedral units into the solution. These units interact and reorganize, forming polymeric chains and networks through the redistribution of oxygen atoms. This reaction results in the development of an amorphous, unstructured GP matrix with strong binding properties [9].

Geopolymer concrete (GC), made from a range of combinations such as metakaolin, silica fume, rice husk ash, fly ash, lime sludge, alccofine, and red mud, has demonstrated exceptional mechanical performance and durability [10-12]. These tailored blends are particularly effective in enhancing strength and reducing porosity, making them ideal for high-performance and high-strength concrete applications, especially in sustainable construction. [13,14] Among these materials, fly ash (FA) stands out due to its widespread accessibility, less water demand, high workability, and rich alumino-silicate composition. In geopolymer synthesis, FA-based concrete gains strength gradually under ambient temperatures (around 25°C). However, to achieve the desired strength within a practical time frame, elevated curing temperatures between 40°C and 75°C are generally required [15]. For effective binding, low-calcium FA is preferred, with a CaO content of less than 5%, Fe_2O_3 content below 10%, a loss on ignition (LOI) under 5%, and reactive silica content in the range of 40% to 50%. Additionally, the fineness of FA is crucial, with 80-90% of particles needing to be smaller than 45 μm [16]. One of the main challenges in using FA-based GC is its slow setting time and delayed strength development, largely due to the less reactivity of FA [17]. To address this issue, researchers have proposed two key strategies. The first strategy involves incorporating calcium-rich industrial wastes, such as ground granulated blast furnace slag (GGBS), flue gas desulfurization gypsum, or small amounts of Portland cement, to accelerate the reaction process. The second approach focuses on mechanically processing fly ash to enhance its reactivity and improve its performance in

geopolymer systems [18]. GGBS, another notable industrial by-product used in GC, is sourced from iron-making plants. This granular material contains more than 35% CaO, over 35% SiO_2 , and less than 15% Al_2O_3 . The incorporation of GGBS in GC plays a crucial role in strength development. During the activation of GGBS, the primary reaction product formed is calcium silicate hydrate (C-S-H) gel, which partially contributes to achieving sufficient strength, even under ambient curing conditions [19]. The synergistic combination of FA and GGBS further enhances the strength and stability of GC. This is attributed to the Al-Si content of FA, which undergoes dissolution, polymerization in the presence of alkali, condensation, and eventual solidification, resulting in a robust and durable matrix [20]. In GC, the silica-to-silica-hydroxide (SS/SH) ratio has been found to significantly influence strength development. Optimal compressive strength (CS) values were observed at SS/SH ratios of 2 and 2.5 for GPC [21,22]. The molar concentration of the alkaline solution is another critical factor. As the molar concentration increases, the compressive strength improves, but only up to a certain threshold. Maximum strength was achieved at molar concentrations between 16 and 18 M, while the minimum compressive strength occurred at 5 M for 7, 14, and 28 days [23]. Furthermore, optimal strength can be attained by carefully adjusting factors such as the liquid-to-binder (L/B) ratio, SS/SH ratio, and binder type. Notably, increasing the L/B ratio up to 0.40 alongside higher molar concentrations further enhances compressive strength [24].

Pond ash (PA), a by-product of coal combustion, has gained recognition as a sustainable material for GC. Similar to fly ash, PA exhibits optimal binding properties when its composition includes low-calcium content (CaO < 5%), Fe_2O_3 below 10%, loss on ignition (LOI) under 5%, and reactive silica content between 30% and 40%. Furthermore, the particle fineness of PA significantly influences its performance, with 80–90% of particles required to be smaller than 45 μm to achieve the desired reactivity [25-28]. Lime sludge (LS), a by-product of the paper industry, also holds promise as a potential GC ingredient, though its utilization remains limited. LS is characterized by a high calcium oxide (CaO) content exceeding 45%, low silicon dioxide (SiO_2) content of less than 15%, and aluminum oxide (Al_2O_3) content below 20% [29]. Despite the limited number of studies on the use of lime sludge-based geopolymer concrete and its impact on strength aspects, the effect of lime sludge addition on the mechanical and microstructural properties of LSGC has yet to be thoroughly explored. In this study, the mechanical properties of GC were evaluated using

various proportions of PA and LS with an 8 M SH solution. The microstructure of the GC was examined using Scanning Electron Microscopy (SEM).

2. MATERIALS AND METHODS

The binders used in this study were pond ash (PA) and lime sludge (LS). PA was sourced from the coal-based Thermal Power Station, Mettur. while LS, a by-product of the paper industry, was obtained from Seshasayee Paper and Boards Ltd., Alampalayam. PA and LS has a specific gravity of 2.17 and 1.96. The chemical compounds of PA and LS of the results were depicted in Table 1.

Table 1. Chemical compounds of LS and PA

Chemical Compounds	LS (%)	PA (%)
CaO	48.5	0.92
SiO ₂	13.27	52.4
Al ₂ O ₃	16.35	30.5
Fe ₂ O ₃	15.06	7.16
MgO	6.65	0.87
SO ₃	0.04	4.28

Sodium silicate (SS) and sodium hydroxide (SH) were used as the activators for the binders, and these chemicals were sourced from local chemical suppliers. Coarse aggregate for the GC was obtained from locally sourced granite, while M-sand was used as the fine aggregate. The coarse aggregate was categorized by passing through 20 mm and 12 mm IS sieves, and the fine aggregates passed through a 4.75 mm IS sieve. The alkaline solution was prepared 24 hours prior to casting the GC. This process involved calculating the required mass of SH to achieve the desired molarity of 8 M, which was then dissolved in a measured quantity of water. The SH solution was blended with a predetermined amount of SS solution. Based on literature study [30,31], the optimal ratio of SS to SH was maintained at 2.5:1, as this ratio has been reported to yield the most favorable results in terms of performance. The GC mix proportions were developed based on prior studies, with a target density of 2400 kg/m³. Detailed mix proportion data for lime sludge-based geopolymer concrete (LSGC) formulations were provided in Table 2.

Table 2. Mix proportions of LSGC

Mix Destination	Binders (%)		Aggregate (kg/m ³)		Alkaline Solution (kg/m ³)		Molarity (M)	L/B Ratio
	PA	LS	Fine	Coarse	Na ₂ SiO ₃	NaOH		
LSGC0	100	-	610	1220	116.28	46.51	8	0.4
LSGC1	90	10	610	1220	116.28	46.51	8	0.4
LSGC2	80	20	610	1220	116.28	46.51	8	0.4
LSGC3	70	30	610	1220	116.28	46.51	8	0.4
LSGC4	60	40	610	1220	116.28	46.51	8	0.4
LSGC5	50	50	610	1220	116.28	46.51	8	0.4
LSGC6	40	60	610	1220	116.28	46.51	8	0.4
LSGC7	30	70	610	1220	116.28	46.51	8	0.4
LSGC8	20	80	610	1220	116.28	46.51	8	0.4
LSGC9	10	90	610	1220	116.28	46.51	8	0.4
LSGC10	-	100	610	1220	116.28	46.51	8	0.4

The LSGC specimens were cured under ambient conditions. The testing of LSGC cubes, LSGC cylinders, and LSGC prisms were depicted in Fig. 1, Fig.2 and Fig.3. Compressive strength tests were conducted using cubes measuring 150 mm on each side at 3, 7, and 28 days curing, in accordance of IS 516 specifications [32]. Similarly, split tensile strength tests were performed on cylindrical specimens with dimensions of (Diameter x Height) 150 mm x 300 mm, following IS 516 specifications at the same curing intervals. Flexural strength was evaluated using prisms with dimensions of (Length x Breadth X Depth) 100 mm x 100 mm x 500 mm, also tested in compliance with IS 516 codes at 3, 7, and 28 days.



Figure 1. Compressive strength test of LSGC

For microstructural analysis, Scanning Electron Microscopy (SEM) images were obtained at Karunya Institute of Technology and Sciences, Coimbatore. Samples for SEM analysis were meticulously selected from fragments of GC specimens post mechanical testing.



Figure 2. Split tensile test of LSGC



Figure 3. Flexural test of LSGC

3. RESULT AND DISCUSSION

3.1. Compressive strength of LSGC

The compressive strength results of lime sludge-based geopolymer concrete (LSGC) mixes were presented in Fig. 4. For the mix LSGC0, comprising 100% pond ash (PA), the compressive strengths noted after 3, 7, and 28 days of ambient curing were 8.13 MPa, 14.04 MPa, and 22.34 MPa, respectively. In contrast, the mix LSGC10, containing 100% lime sludge (LS), demonstrated significantly higher strengths than LSGC0 mix, achieving 10.88 MPa, 22.11 MPa, and 33.71 MPa for the same curing durations. Among all the mixes, LSGC5 mix exhibited the highest compressive strengths, recording 27.98 MPa, 41.44 MPa, and 53.32 MPa at 3, 7, and 28 days of curing, respectively, outperforming all other compositions. This superior performance can be attributed to the optimal balance of PA and LS, which facilitated the enhanced formation of calcium-alumino-silicate-hydrate (C–A–S–H) gel, significantly improving the mechanical properties of the GC. The combined use of PA and LS contributed to superior strength due to their high alumina-silicate content and larger specific surface area, enabling the formation of additional geopolymeric gel. Additionally, the silica and free lime present in PA and LS enriched the C–A–S–H gel, further strengthening the concrete. However, mixes with excessively high proportions of PA or LS exhibited incomplete geopolymerization reactions, likely due to insufficient alkaline content. Similar reports were found by [33,34]. This deficiency impeded effective polymerization and resulted in reduced compressive strength. Similarly, the LSGC0 mix showed inadequate strength development due to the low calcium oxide (CaO) content in PA, which was insufficient to activate effective polymerization under ambient curing conditions. Similar result was identified by [35].

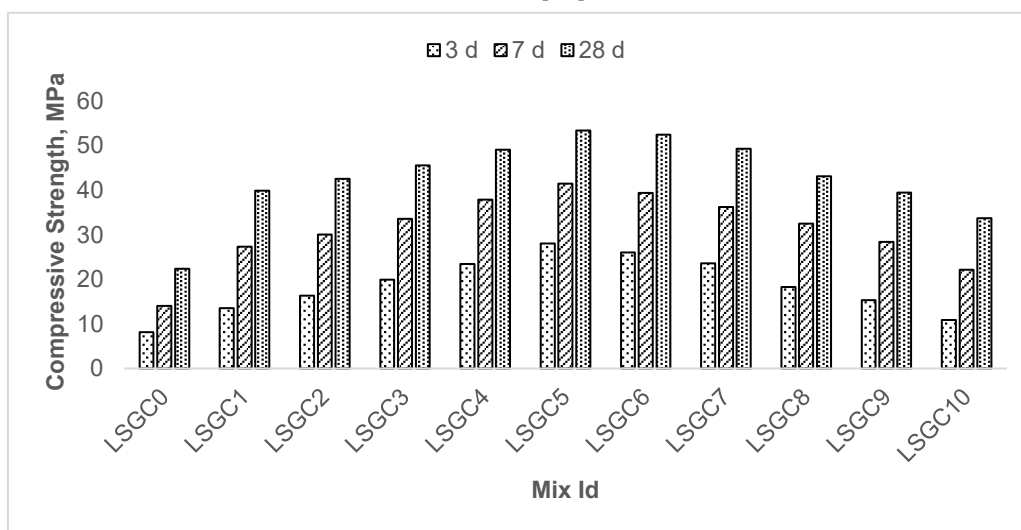


Figure 4. Compressive strength results of LSGC mixes

3.2. Split tensile strength of LSGC

The split tensile strength results of lime sludge-based geopolymer concrete (LSGC) mixes were presented in Fig. 5. For mix LSGC0, consisting of 100% pond ash (PA), the split tensile strengths noted after 3, 7, and 28 days of ambient curing were 1.33 MPa, 1.84 MPa, and 2.34 MPa, respectively. In comparison, mix LSGC10, comprising 100% lime sludge (LS), exhibited significantly higher tensile strengths, achieving 1.43 MPa, 2.21 MPa, and 3.51 MPa for the same curing durations, clearly surpassing the performance of LSGC0 mix. Among all the compositions, mix LSGC5 achieved the highest split tensile strengths, with values of 2.68 MPa, 4.44 MPa, and 5.32 MPa at 3, 7, and 28 days, respectively, outperforming other mixes. The enhanced performance of LSGC5 mix can be attributed to the optimal combination of PA and LS, which facilitated improved polymerization and strength development under ambient curing conditions. The poor performance of

100% PA-based GC (LSGC0 mix) is likely due to its inability to achieve sufficient strength without an external energy source. Pure PA-based GC typically requires a minimum of 24 hours of oven curing at elevated temperatures to initiate polymerization and achieve satisfactory strength. However, introducing lime sludge (LS) into the mix eliminates the need for oven curing by enabling effective polymerization and strength gain at ambient temperatures. As evident from Fig. 5, the tensile strength increased progressively with higher LS content in the mix. However, the optimal performance was observed at a 50% addition of LS with PA, particularly at 28 days of ambient curing. This composition achieved superior strength due to the enhanced formation of geopolymeric and C–A–S–H gels, resulting in improved mechanical properties. Excessive LS content beyond the optimal proportion, however, did not yield additional strength benefits. Similar results were reported by various researchers [36,37].

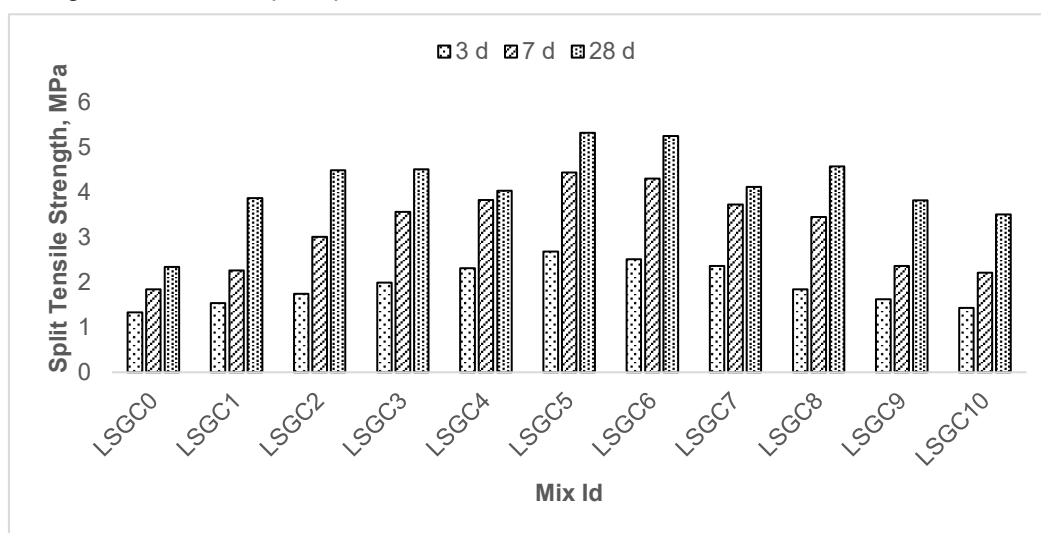


Figure 5. Split tensile strength results of LSGC mixes

3.3. Flexural strength of LSGC

The flexural strength results of lime sludge-based geopolymer concrete (LSGC) mixes were presented in Fig. 6. For mix LSGC0, consisting of 100% pond ash (PA), the flexural strengths noted after 3, 7, and 28 days of ambient curing were 2.73 MPa, 3.64 MPa, and 4.57 MPa, respectively. In comparison, mix LSGC10, comprising 100% lime sludge (LS), exhibited significantly higher tensile strengths, achieving 3.25 MPa, 4.57 MPa, and 5.64 MPa for the same curing durations, clearly surpassing the performance of LSGC0 mix. Among all the compositions, mix LSGC5 achieved the highest flexural strengths, with values of 5.15 MPa, 6.27 MPa, and 7.08 MPa at 3, 7, and 28 days of curing, respectively, outperforming other mixes.

Except for 100% PA based mixes of GC remaining, all shown good results. However, the highest performance was observed at a 50% addition of LS with PA, particularly at 28 days of ambient curing. The 50% of LS and PA in GC, enhancement of flexural strength due to fine PA and LS, which filled up the pores and leads to make dense matrix of GC.

The improved flexural strength can be attributed to the ultra-fine particles of PA and LS, which offer a larger surface area, facilitating the polymerization process. This promotes the formation of additional geopolymeric gel, enhancing cohesion between aggregates and ultimately increasing strength. Similar trend was observed by [38,39].

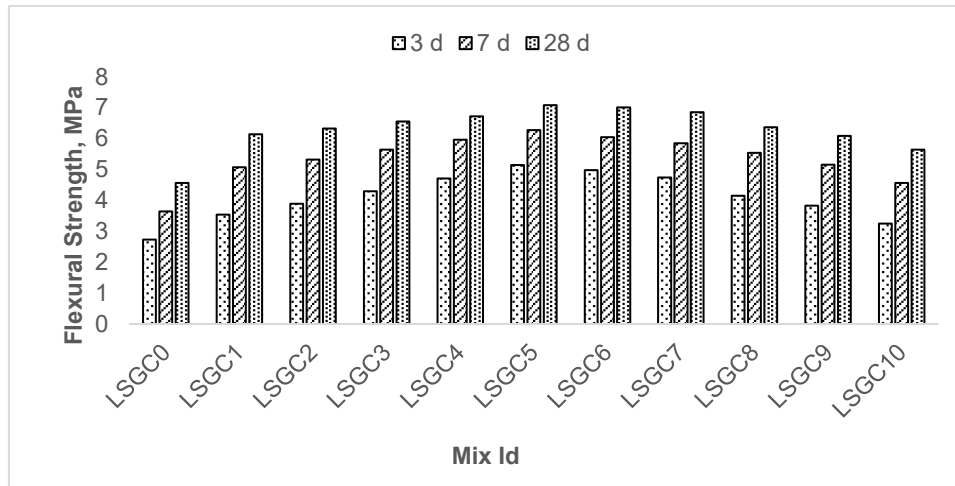


Figure 6. Flexural strength results of LSGC mixes

3.4. Correlative analysis on mechanical properties of LSGC

A correlation can be established between the compressive strength (f_c) and split tensile strength (f_{sts}), and compressive strength (f_c) and flexural strength (f_s) of the GC mixes. As shown in Fig. 7, the f_{sts} were approximately 0.17 root mean square of f_c values. Similarly, the f_s were found to be 0.96 times root mean square of f_c (Fig. 8).

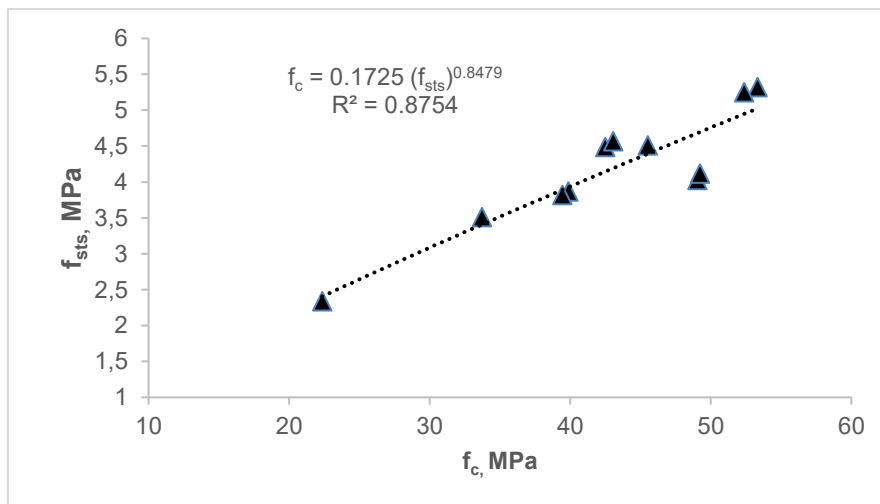


Figure 7. Predicted f_{sts} of LSGC

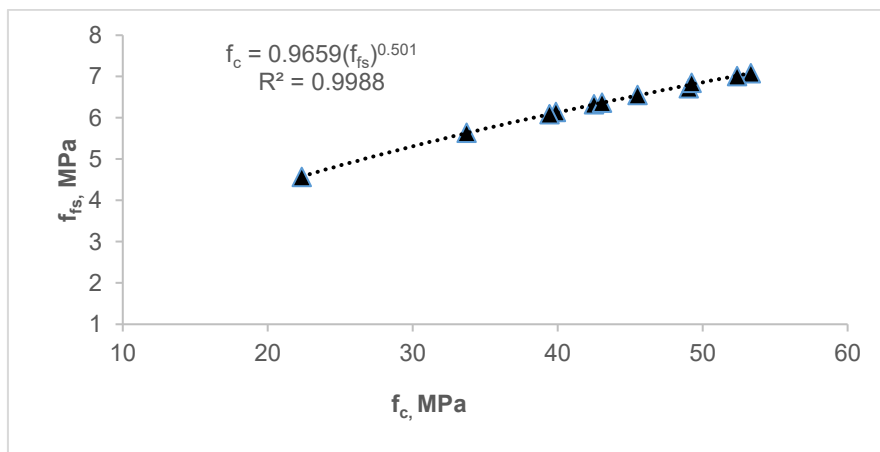


Figure 8. Predicted f_s of LSGC

3.5. SEM analysis of LSGC

The enhancement in LSGC mixes strength were analyzed through SEM analysis. Two mix compositions, LSGC0 (100% PA) and LSGC5 (50% PA and 50% LS), were selected for microstructural examination after 28 days of curing. The SEM images, presented in Fig. 9, reveal distinct differences between the mixes. In the LSGC0 mix, a significant portion of angular fragment particles of PA and the presence of pores were evident. This incomplete geopolymerization, along with the absence of a dense microstructure, was identified as the primary reason for the lower strength attainment in LSGC0 than LSGC5 mix. Conversely, the SEM analysis of the LSGC5 mix indicated a

highly reactive index and a significantly denser microstructure. The micrographs demonstrated the formation of C–A–S–H gel, predominantly resulting from the interaction of LS with PA. The higher calcium content provided by LS contributed to the formation of additional binding agents, thereby enhancing the hardening properties of the geopolymer. The densely packed microstructure observed in LSGC5 mix was indicative of enhanced geopolymeric gel formation, which resulted in superior strength characteristics. This structural enhancement highlights the pivotal role of LS in improving the mechanical and microstructural properties of GC through the formation of supplementary C–A–S–H seeds and a cohesive matrix.

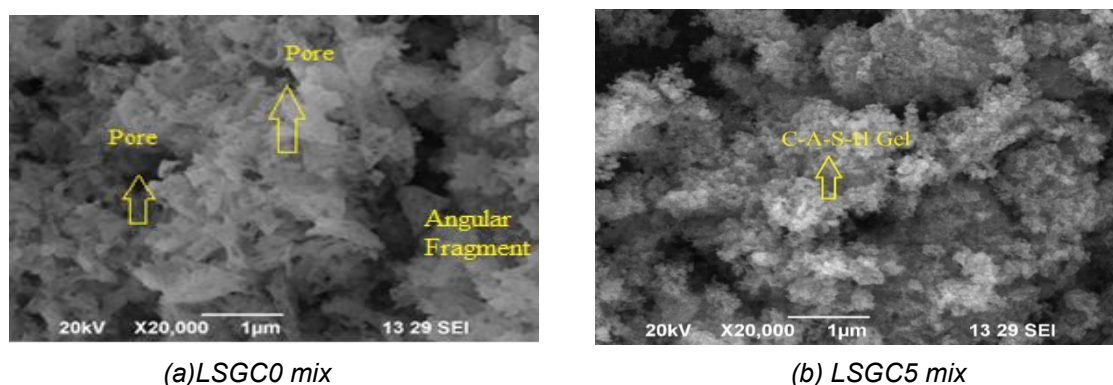


Figure 9. Microstructural properties of (a) LSGC0 mix and (b) LSGC5 mix

4. CONCLUSION

The following conclusions were drawn on GC mixes with and without the addition of LS and given below

- The LSGC5 mix, consisting of 50% PA and 50% LS, achieved the highest compressive strength values of 27.98 MPa, 41.44 MPa, and 53.32 MPa at 3, 7, and 28 days, respectively. For split tensile strength, the LSGC5 mix demonstrated strengths of 2.68 MPa, 4.44 MPa, and 5.32 MPa at 3, 7, and 28 days. Its flexural strength reached 5.15 MPa, 6.27 MPa, and 7.08 MPa at 3, 7, and 28 days.
- This optimal LSGC5 mix outperformed all other compositions, benefiting from the balanced interaction between PA and LS, which facilitated the formation of C–A–S–H gel seeds and significantly improved mechanical properties. The inclusion of LS promoted polymerization even under ambient curing conditions, addressing the limitations faced by pure PA-based mixes, which struggled to achieve strength without external thermal curing.
- A correlation analysis showed that the split tensile strength (fst) was approximately 0.17 times the square root of compressive strength (fc), while the flexural strength (ff) was about

0.96 times the square root of compressive strength.

- SEM analysis revealed that the microstructure of the LSGC5 mix exhibited enhanced densification and higher reactivity compared to the LSGC0 mix. In contrast, the LSGC0 mix had angular fragment structure and significant porosity, leading to lower strength. The LSGC5 mix, however, demonstrated a denser matrix enriched with supplementary C–A–S–H gel, which contributed to its superior mechanical and microstructural properties.

Acknowledgements

The authors wish to acknowledge Department of Civil Engineering, K.S.Rangasamy College of Technology, Tiruchengode for the facility and support extended for the research work.

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IZVOD

UTICAJ DODAVANJA KREČNOG MULJA NA MEHANIČKA I MIKROSTRUKTURNA SVOJSTVA GEOPOLIMER BETONA NA BAZ PEPELA

Ova studija istražuje dodavanje različitih proporcija krečnog mulja (LS) i trajanja očvršćavanja (3, 7 i 28 dana) na svojstva geopolimer betona (GC). Sve GC mešavine su pripremljene sa 8 M rastvorom natrijum hidroksida i fiksnim odnosom tečnosti prema vezivu od 0,4. Procenjene su mehaničke osobine mešavina GC na bazi LS, a zatim kontrolna mešavina koja se u potpunosti sastojala od pepela iz ribnjaka (100% PA). Pored toga, izvršena je analiza skenirajuće elektronske mikroskopije (SEM) da bi se istražile mikrostrukturne razlike između kontrolne mešavine (100% PA, LSGC0) i optimizovane mešavine sa 50% PA i 50% LS (LSGC5). Rezultati su otkrili da je mešavina LSGC5 postigla značajna poboljšanja u čvrstoći na pritisak, rascepu zatezanja i savijanja u poređenju sa mešavinom LSGC0. SEM analiza je istakla formiranje gušće i kohezivnije mikrostrukture u mešavini LSGC5, što se pripisuje povećanom stvaranju gelova kalcijum-alumino-silikat-hidrata (C–A–S–H), koji su bili manje zastupljeni u mešavini 100% PA. Ovi nalazi demonstriraju efikasnost krečnog mulja kao održivog zamenskog materijala, značajno poboljšavajući mehanička i mikrostrukturna svojstva GC-a uz smanjenje zavisnosti od tradicionalnih cementnih komponenti.

Ključne reči: Geopolimer beton; ribnjak pepeo; krečni mulj; mehanička svojstva; mikrostrukturna svojstva.

Naučni rad

Rad primljen: 08.12.2024.

Rad korigovan: 24.01.2025.

Rad prihvaćen: 28.01.2025.

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