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Investigation of Compressive Strength of Slag-Based Geopolymer Concrete Incorporated with Rice Husk Ash Using 12M Alkaline Activator

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***Abstract*—**Geopolymer concrete's (GPC) compressive strength was investigated. The GPC was incorporated with rice husk ash (RHA) and ground granulated blast furnace slag (GGBFS), which may have potential in the construction industry to replace Portland limestone cement (PLC) concrete. The sustainable construction binders used were GGBFS and RHA, and a solution of sodium hydroxide (NaOH) and sodium silicate gel (Na_2SiO_3) was used as the 12-molar alkaline activator. Five GPC mixes comprising fine aggregates, coarse aggregates, GGBS, and RHA, and the alkaline solution in the ratio 2: 2.5: 1: 0.5, respectively, were prepared to achieve grade 40 concrete, and PLC was substituted with GGBFS and RHA in the ratios of 0:100, 25:75, 50:50, 75:25, and 100:0. A control mix was also prepared which comprised of 100% water and 100% PLC as the cementitious material. The GPC mixes were thermally cured at 60-80 ºC in an oven for approximately 24 h. After curing for 7 and 28 days, the compressive strength test results of the hardened GPC samples showed that GPC-Mix #3, comprising 50% GGBFS and 50% RHA, was the most efficient geopolymer mix. The mix had compressive strengths of 35.71 MPa and 47.26 MPa, 19.87% and 8.69% higher than the PLC concrete samples, which had 29.79 MPa and 43.48 MPa after 7 and 28 days, respectively. Therefore, GPC containing GGBFS incorporated with RHA is an efficient method of decreasing the use of PLC in conventional concrete production and reducing the high amounts of CO_2 emitted into the atmosphere in the construction industry.

***Keywords*—**Alkaline solution, cementitious material, geopolymer concrete, ground granulated blast furnace slag, rice husk ash.

I. INTRODUCTION

MORE structural and infrastructural facilities are being developed due to globalization and industrialization, leading to a greater demand for concrete use [1]. This increased the production of PLC, the chief constituent of concrete, to accommodate the need for shelter and economic activities. However, there are many difficulties related to producing PLC: it requires a large quantity of energy, depletes the ozone layer, and pollutes the environment, as carbon dioxide is usually released [2]. Hence, developing alternatives to conventional cement production is essential to eliminate or reduce the challenges of producing PLC.

References [3]-[7] have proved that waste materials containing aluminum (Al) and silicon (Si), such as metakaolin, fly ash, silica fume, GGFBS, and RHA, can improve mortar and concrete's durability and strength. Reference [8] also investigated the polymerization reaction between an alkaline solution and a source material containing aluminum (Al) and silicon (Si). The results proved that geopolymer or mineral binders are products of geopolymerization reactions. In support, [9] opined that the two main constituents of mineral binders control their properties. Further, [10] reported that there are benefits of producing GPC over PLC. These benefits include low production costs and energy-efficient production processes.

The primary binder in PLC is calcium-silicate hydrates, unlike in GPC, where it is an alumino-silicate polymeric gel formed by tetrahedral-bonded silicon and aluminum with oxygen atoms shared in between. Many researchers like [27] and [43] have also shown that adding GGBFS, which contains soluble silicates in large amounts, to the geopolymeric gel leads to calcium dissolution at low alkalinity, forming C-S-H gel together with the geopolymeric gel. Consequently, the coexistence of the C-S-H and geopolymeric gels improves the mechanical strength of GPC [11].

Reference [12] stated that RHA could be a source and supplemental cementitious material in GPC with GGBFS. The authors' experimental investigations on the partial replacement of cement with RHA found that the RHA improved the strength of the concrete and increased its durability. Although early compressive strength was slightly less in RHA concrete than in conventional concrete, it was observed that at 7.5% replacement, the RHA concrete displayed higher compressive strength when compared to the conventional concrete. Further research found that during compressive strength testing on various concrete mixes with varying RHA percentages by [13], 20% RHA was the optimum dosage level for all their mixes.

Concerning workability, the RHA samples were very porous due to their large specific surface area, resulting in decreased workability in the concrete as the RHA samples consumed excessive water. However, adding commonly used chemical admixtures such as plasticizers can achieve the desired workability [13].

Reference [14] studied the properties (durability and strength) of GPC using black rice husk ash (BRHA) and

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GGBFS, whereby GGBFS replaced BRHA at proportions of 10, 20, and 30%. The results revealed that the GPC's strength increased with increasing curing temperature. However, they discovered that compressive strength development was stunted with BRHA beyond 10%. Nevertheless, the compressive strength surpassed the target strength by up to 20% replacement levels.

The curing of PLC concrete is done in water or an ambient environment. Conversely, GPC is cured thermally between 60-80 °C to achieve optimum compressive strength [15], [16]. In addition, [17] discovered that GPC would attain its optimum compressive strength at 60 °C. During thermal curing, the water usually produced during the polymerization process is vaporized. Thus, the GPC hardens, its compressive strength significantly increases, and drying shrinkage is minimized as the water vaporizes. GPC's compressive strength also increases with the curing period. However, 20 hours is sufficient for achieving optimum compressive strength, as curing beyond 24 hours does not significantly increase GPC's compressive strength [18].

This research investigated GPC's compressive strength produced with varying proportions of GGBFS and RHA as binders, with GGBFS and RHA in the ratios of GGBFS(0): RHA(100), GGBFS(25):RHA(75), GGBFS(50):RHA(50), GGBFS(75):RHA(25), and GGBFS(100):RHA(0). This study also assessed the sustainability of using GPC utilizing GGBFS and RHA.

II. MATERIALS AND METHODS

A. Materials

The materials used are RHA, GGBFS, coarse aggregates, fine aggregates, water, PLC, sodium silicate (Na_2SiO_3), and sodium hydroxide (NaOH). The sodium hydroxide pellets and liquid sodium silicate were supplied by a chemical and laboratory equipment store in Ibadan, Nigeria, and 42.5R PLC from a local cement store in Ibadan, Nigeria. The GGBFS used was sourced from a steel company in Ogun State, Nigeria. Natural river sand was used as fine aggregate, and both the fine and coarse aggregates (19 mm angular and well graded) were sourced locally in Ibadan, Nigeria. The RHA was also sourced locally from a rice mill in Akure, Ondo, Nigeria.

B. Materials Characterization

1. Chemical Compositions

The RHA, GGBFS, and PLC materials consist of oxides. These oxides were determined via X-ray fluorescence (XRF) spectrometry analysis. Table I shows the analysis result.

References [19] and [20] state that suitable pozzolanic binder materials must have 70% of their constituents consisting of SiO_2, Al_2O_3, and Fe_2O_3. In this study, the RHA material met this criterion. It had its SiO_2, Al_2O_3, and Fe_2O_3 constituents equal 83.07%. Consequently, the RHA material was suitable for GPC production. Reference [21] also used an RHA material of similar composition in their study.

According to [22], a suitable GGBFS material for GPC production must have 32-40% SiO_2 and CaO contents. The GGBFS material met this requirement, containing 35.77% SiO_2 and 36.52% CaO. Thus, it is suitable for GPC production. References [23] and [24] also used a GGBFS material of similar composition in their study. The PLC used for producing the control samples also met the criteria laid out in [25].

TABLE I
RHA, GGBFS, AND PLC CHEMICAL COMPOSITIONS

Oxides	RHA (%)	GGBFS (%)	PLC (%)
SiO_2	79.94	35.77	21.60
Al_2O_3	1.82	14.11	5.85
Fe_2O_3	1.31	0.92	2.78
CaO	1.65	36.52	64.30
MgO	1.85	9.45	1.42
Na_2O	2.60	0.30	0.14
K_2O	2.54	0.52	0.72
SO_3	1.17	1.08	2.03
Loss on Ignition (LOI)	4.22	1.45	1.38

2. Microstructural Behaviors

The Scanning Electron Microscopy (SEM) images of RHA, GGBFS, and PLC are shown in Figs. 1 (a)-(c). From the SEM images, RHA has irregularly shaped particles with rough, porous, and large surfaces, while uneven surfaces and amorphous shapes characterize GGBFS. Likewise, angular shapes characterize PLC, having a somewhat wrinkled internal structure. As a result, GGBFS and RHA's surface area, shape, and structure greatly control the properties of GPC [26].

C. RHA Preparation

After sourcing and collecting rice husks, the husks were burnt in batches in the open air to ashes, and undesirable particles were removed from the resulting material by sieving with a 300 μm sieve. After that, calcination was conducted in a closed furnace for 5-6 h. It was ensured that the furnace's temperature did not exceed 600 °C while calcinating the RHA material. Further, the calcinated RHA was left to cool for 24 hours, as shown in Fig. 2. Afterward, the ashes were ground by a miller to obtain the most refined form of ash with no particle size beyond 45 μm.

D. Preparation of Alkaline Solution

According to Kumar [27], the alkaline solution must be prepared before casting the GPC test samples. References [14] and [28] also suggested a ratio of 1: 2.5 for preparing the alkaline activator. Thus, the 12M alkaline solution preparation was 24 hours prior with industrial grade NaOH and Na_2SiO_3 in the ratio 1: 2.5.

E. Geopolymerization Reaction and Mechanism

Geopolymerization refers to the rapid chemical reaction occurring when an alkaline solution reacts with a source material containing aluminosilicate [29]. This process forms a three-dimensional polymeric chain and ring structure characterized by Si-O-Al-O bonds [29]. The resulting product from a typical geopolymerization process can be represented by the composition formula $nM2O \circ Al_2O_3 \circ xSiO_2 \circ yH_2O$, where "M" represents an alkaline element, and "n" denotes the degree of polycondensation [30].

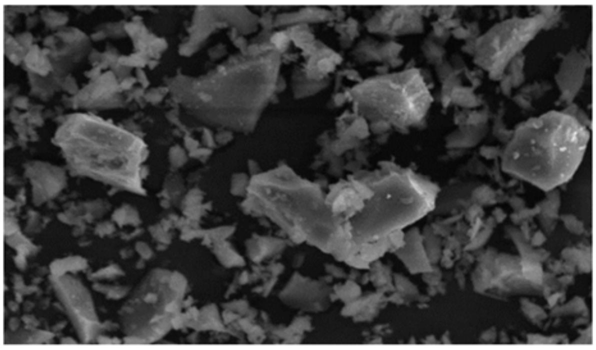


Fig. 1 (a) SEM of RHA

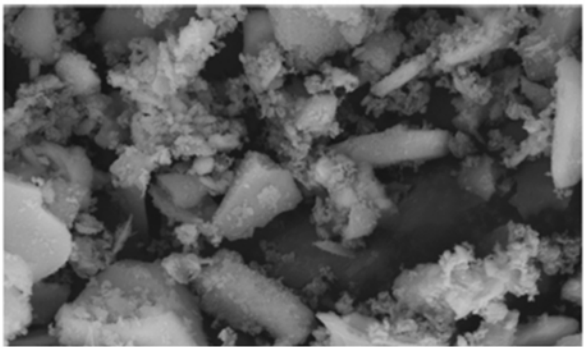


Fig. 1 (b) SEM of GGBFS

Fig. 1 (c) SEM of PLC

Fig. 2 Calcinated and milled RHA

The Si to Al ratio plays a crucial role in determining the ultimate structure of a geopolymer. Geopolymer materials with a Si to Al ratio ranging from 2 to 3.5 exhibit highly rigid structures, making them ideal for various construction projects [31]. Further, [31] identified three distinct types of polysialates found in geopolymers: polysialates (-Si-O-Al-O-), polysiloxo sialates (-Si-O-Al-O-Si-O-), and poly-disiloxo sialates (-Si-O-Al-O-Si-O-Si-O). For a visual representation of these three polysialates, please refer to Fig. 3.

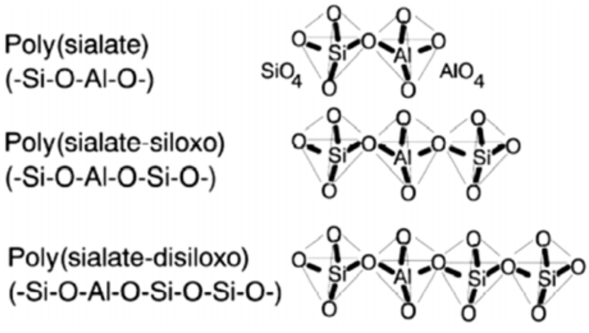


Fig. 3 Polysialates chemical structures [32]

According to [33], the formation mechanism of geopolymer material during setting and hardening involves a series of chemical reactions that can be described by the following three steps, as represented by (1), (2), and (3) in Fig. 4:

1) The alkaline solution initiates the dissolution of Si and Al atoms.
2) The dissolved ions undergo reorganization and diffusion, forming monomers.
3) The monomers then undergo polymerization, resulting in the formation of polymeric or hydrated products.

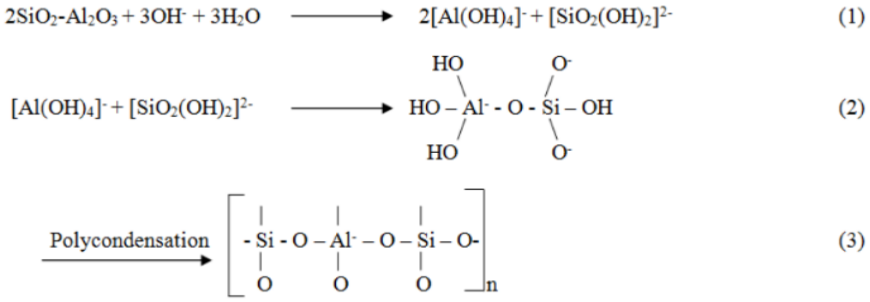
During the curing process of GPC, water is typically released. In contrast, PLC concrete absorbs water through hydration. Consequently, the disparity in curing processes, particularly the application of heat to facilitate polymerization in GPC, significantly affects its mechanical and chemical properties. It influences its resistance to chemical attacks, water ingress, alkali-aggregate reactivity, and heat [32].

F. Life Cycle Analysis of Materials

GPC presents a highly efficient alternative to PLC concrete due to its remarkable durability properties, resistance against sulphate and acid attacks, higher mechanical strength, and lower heat of hydration [1]. In addition to these advantages, the production of GPC also yields several beneficial outcomes, including reduced waste disposal in landfills, conservation of natural resources, and environmental preservation [14].

Agricultural and industrial processes generate substantial waste materials such as RHA and GGBFS, typically disposed of in landfills. However, these waste materials can be utilized effectively as cementitious components in GPC production [34], [35].

It is important to note that the production of PLC concrete results in the emission of significant amounts of CO_2. For every 1 kg of PLC produced, approximately 1 kg of CO_2 is released into the environment [1]. Reference [8] elaborated on this one-to-one ratio by explaining that 1 ton of PLC generates 0.55 tons of CO_2, and an additional 0.40 tons is produced during carbon fuel combustion. Thus, developing and adopting sustainable alternatives are crucial for mitigating environmental pollution challenges and other health hazards.



$$2SiO_2\text{-}Al_2O_3 + 3OH^- + 3H_2O \longrightarrow 2[Al(OH)_4]^- + [SiO_2(OH)_2]^{2-} \quad (1)$$

$$[Al(OH)_4]^- + [SiO_2(OH)_2]^{2-} \longrightarrow \quad (2)$$

Fig. 4 The geopolymerization process in three steps [33]

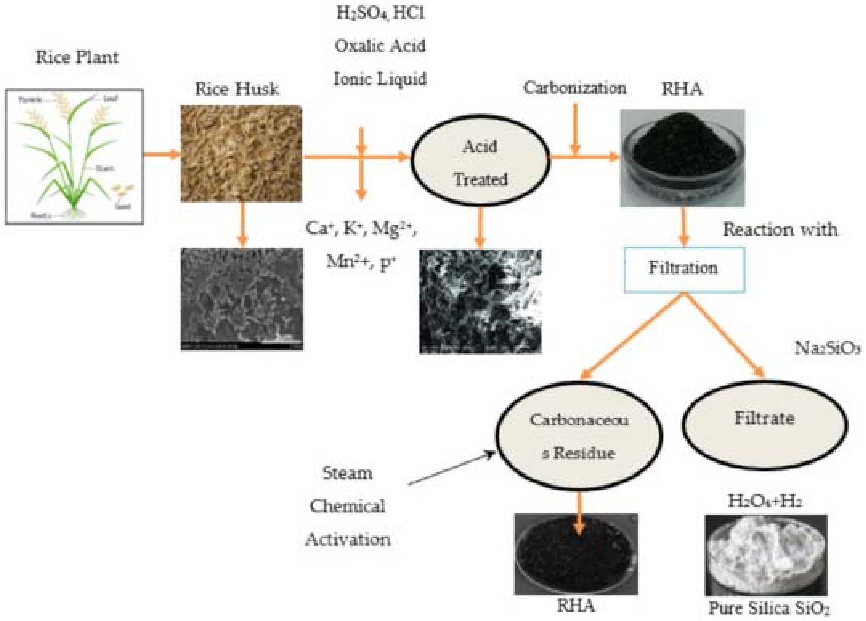


Fig. 5 Source and Pre-treatment of RHA [39]

1. Life Cycle Analysis of RHA

Fig. 5 shows the source and pretreatment of RHA. Rice husk is the main by-product containing approximately 50%, 25-30%, and 15-20% of cellulose, lignin, and silica contents, respectively [36]. Rice husk is a waste product generated during rice milling and is often disposed into waterways, leading to their contamination and pollution [37]. Thus, it is available in large quantities in many countries. About 20% of rice husk is produced, with 25% becoming RHA after burning for every milled paddy. As a result, above 75% of silica and RHA are left when rice husk is burnt under controlled temperature and environment [38].

Reference [37] reported that previous studies on the production of RHA found that the process is energy-efficient and produces low emissions compared to other combustion processes. In contrast, [40] reported that RHA has generally produced additional criteria air pollutants and global warming potential compared to fly ash because it requires higher amount of energy for grinding. Further, using RHA in various applications can provide environmental benefits, such as reducing the use of virgin materials, improving soil fertility, and reducing greenhouse gas emissions [41]. However, assessing the environmental impacts of using RHA is vital, such as the nitrous oxide emissions associated with its application as a soil stabilizer.

Finally, the disposal of RHA can also have environmental impacts, such as landfilling or incineration. To solve this challenge, several studies have suggested that RHA can be used as a raw material in the production of ceramics, glass, and greener and structural concrete, thus reducing the need for virgin materials and promote a circular economy [37], [39], [21].

2. Life Cycle Analysis of GGBFS

Global slag production exceeds 400 million tons annually, as reported by [42]. Therefore, extensive research has been conducted on slag as a means of steel production residues recycling and creating resource-efficient, low-carbon pozzolanic binders for concrete production [43]. Slag, a widely used by-product of iron and steel manufacturing, is formed by

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rapidly cooling molten slag from a blast furnace using water, steam, or a combination of both. This process results in a glassy granular substance ground into a fine powder known as GGBFS [44].

Reference [43] demonstrated that carbonation and alkali activation could activate the binding properties of GPC. Hence, utilizing GGBFS through alkali activation and carbonation can help produce new cementitious materials and solve the environmental challenges and threats of the cement and concrete industries. Reference [45] confirmed that using GGBFS in construction does not pose environmental risks and presents a viable and sustainable alternative for repurposing the vast amounts of slag deposited in landfills. Additionally, GGBFS production results in a significant reduction of approximately 80% in CO_2 emissions compared to PLC [8].

Further, the study indicated that the energy required to produce 1 tonne of PLC is approximately 4700 MJ of electric power. In contrast, the energy requirements for GGBFS as a steel by-product are significantly reduced by 59%. Therefore, utilizing GGBFS can effectively address the comprehensive environmental issues associated with producing PLC while promoting the conservation of land resources, energy, and materials. However, it is vital to note that slag production still has notable environmental impacts on global warming and acidification potential [46].

G. Test Samples Mix Design

Table II shows the mix of the control PLC samples that resulted in 40 MPa after performing various trial mixes. Unlike the PLC control mix sample, 5 others were prepared where % GGBFS was substituted with RHA. Thus, RHA replaced 0%, 25%, 50%, 75%, and 100% GGBFS in GPC-Mix #1, GPC-Mix #2, GPC-Mix #3, GPC-Mix #4, and GPC-Mix #5, respectively. The quantity of each constituent of the GPC mix per 100 mm cube test sample is presented in Table III.

TABLE II
GPC GRADE 40 MIX DESIGN

Constituents	Ratio	Quantity
Fine Aggregates (CA)	2.0	0.8 kg
Coarse Aggregates (FA)	2.5	1.0 kg
PLC/RHA and GGBFS	1.0	0.4 kg
Water/Alkaline Solution	0.5	200 ml

TABLE III
GPC MIX PROPORTION

Mixes	CA (kg)	FA (kg)	GGBFS (%)	GGBFS (kg)	RHA (%)	RHA (kg)	NaOH (ml)	Na_2SiO_3 (ml)
1	1	0.8	100	0.4	0	0	57	143
2	1	0.8	75	0.3	25	0.08	57	143
3	1	0.8	50	0.2	50	0.16	57	143
4	1	0.8	25	0.1	75	0.24	57	143
5	1	0.8	0	0	100	0.32	57	143

H. Casting of GPC and PLC Samples

The inner surfaces of the molds were cleaned and lubricated with oil prior to mixing and casting the test samples. The coarse aggregates, cementitious materials (cement, GGBFS, or RHA), and fine aggregates, in that order, were weighed and placed in a tray according to the number of cubes being cast, mix ratio, and mix number, as stated in Table II. The sandwich method was employed, which involves having the cementitious material between the coarse and fine aggregates. This method is the most efficient because it helps achieve a homogenous mix while reducing errors.

Further, a measuring cylinder was used to measure the required alkaline solution volume. After measuring the alkaline solution of sodium hydroxide and sodium silicate, the solution was thoroughly mixed with other constituents until a homogenous fresh concrete mix was formed. Subsequently, the resulting fresh concrete was placed in the molds in 3 layers. Each layer was given sufficient tamping blows to achieve maximum compaction and eliminate air voids. After vibrating and thus compaction, the cubes were placed in the curing room at a temperature of 21 °C. Lastly, the cubes were 24 hours to set and harden.

I. Curing of Test Samples

The test samples were given a rest period of 24 hours after casting. The rest period was essential for proper polymerization and improvement of the GPC's mechanical properties. The cubes were then demolded and placed in an oven for thermal curing at a temperature range between 57 °C and 63 °C for another 24 hours, after which the cubes were allowed to cool to room temperature in the oven to avoid a sudden temperature change. After a few hours of cooling in the oven, the cubes were removed and further cooled at room temperature (approximately 30 ºC).

J. Testing of Samples

After curing for 7 and 28 days, two tests were conducted on the PLC and GPC samples: density and compressive strength tests. The British Standard method for determining the compressive strength of 100 mm concrete cubes was employed to determine the test samples' compressive strength [47]. Fig. 6 shows the placement of a test cube in the compressive strength-testing machine. Afterward, the machine applied loads on the samples, and the failure load and compressive strength were recorded for each cube at the point deformation occurred.

III. RESULTS AND DISCUSSION

The average density and compressive strength test results are presented for the control PLC sample mix and each GPC sample mix. The optimum percentage substitution of GGBFS and RHA was also recommended.

Fig. 6 Compressive strength testing of samples

A. Density Test

The PLC and GPC samples were cured for 7 and 28 days, after which their densities were tested. The results are shown in Table IV. Fig. 7 also shows the relationship between the average density of GPC mixes and their RHA content. The average density of GPC-Mix #1 to #3 increased as their RHA content increased, while the average density of GPC-Mix #4 and #5 decreased with further RHA content increment. The average density of GPC-Mix #1 to #5 after 7 days of curing was 3.01%, 3.13%, 5.56%, 1.65% and 1.15% higher than the PLC control mix's average density. However, it was 2.36%, 2.93%, 5.30%, 1.55%, and 1.14% higher at 28 days of curing.

The test samples' density was studied because it is an essential property of concrete, which controls concrete's mechanical properties. A denser concrete would quickly attain fewer voids and increased durability and strength than less dense concrete. Such concrete would also have reduced permeability and low water absorption. Thus, the density values explain why GPC-Mix #3 has the highest compressive strength among the GPC samples containing RHA. The GPC samples are normal-weight concrete as their densities are within 2200-2600 kg/m^3 at 28 days of curing, which shows that the use of GGBFS and RHA does not affect the concrete's density.

TABLE IV
AVERAGE DENSITY OF SAMPLE MIXES

Mixes	7 days average density (kg/m^3)	28 days average density (kg/m^3)
PLC-Mix	2427	2455
GPC-Mix #1	2500	2513
GPC-Mix #2	2503	2527
GPC-Mix #3	2562	2585
GPC-Mix #4	2467	2493
GPC-Mix #5	2455	2483

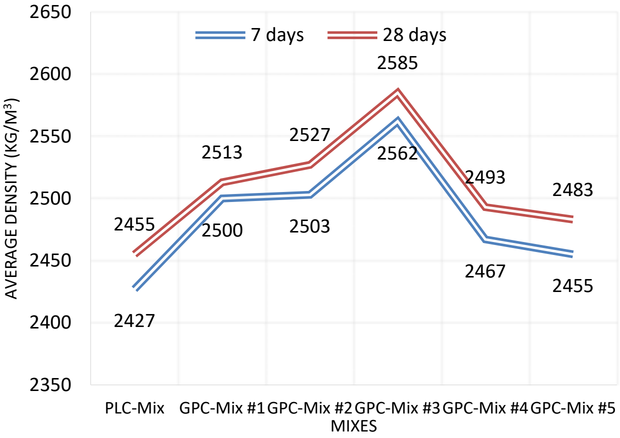

Fig. 7 7- and 28-days average density of sample mixes

B. Compressive Strength Test

The compressive strength of the sample mixes was tested, and the results are shown in Table V. Fig. 8 also compares the sample mixes' average compressive strength. The average compressive strength increased for the first three mixes with 100% GGBFS – 0% RHA, 75% GGBFS – 25% RHA, and 50% GGBFS – 50% RHA. Conversely, it decreased for 25% GGFBS – 75% RHA and 0% GGBFS – 100% RHA. The average compressive strength of GPC-Mix #1 and #3 after 28 days of curing was 8.69% and 2.18% higher than the PLC control mix's average compressive strength. However, the average compressive strength of GPC-Mix #2, #4, and #5 was 6.14%, 21.64%, and 46.78% below the PLC control mix's average compressive strength.

The results show that GPC-Mix #1 and #3 would perform better under compressive loading than the control PLC mix without any crack or deflection. Therefore, GPC-Mix #1 and GPC-Mix #3 suit chemical and thermal-resistant commercial and industrial structures. Conversely, GPC-Mix #2, #4, and #5 are suitable for domestic use.

TABLE V
AVERAGE COMPRESSIVE STRENGTH OF SAMPLE MIXES

Mixes	7 days average compressive strength (MPa)	28 days average compressive strength (MPa)
PLC-Mix	29.79	43.48
GPC-Mix #1	33.08	44.43
GPC-Mix #2	30.37	40.81
GPC-Mix #3	35.71	47.26
GPC-Mix #4	21.71	34.07
GPC-Mix #5	15.91	23.14

C. Optimum Percentage Substitution of GGBFS and RHA

GPC-Mix #1 had the highest average compressive strength after 7 and 28 days. However, the mix does not contain RHA; thus, 0% RHA and 100% GGBFS cannot be considered the optimum substitution of GGBFS and RHA. Figs. 4 and 5 show that the GGBFS-RHA-based GPC attained its highest average compressive strength (44.43 MPa at 28 days), containing 50% RHA and 50% GGBFS with an average density of 2585 kg/m^3. Therefore, it was determined that GPC Mix #3 of 50% RHA and 50% GGBFS at 12M alkaline solutions is the ideal mix for the most efficient concrete production.

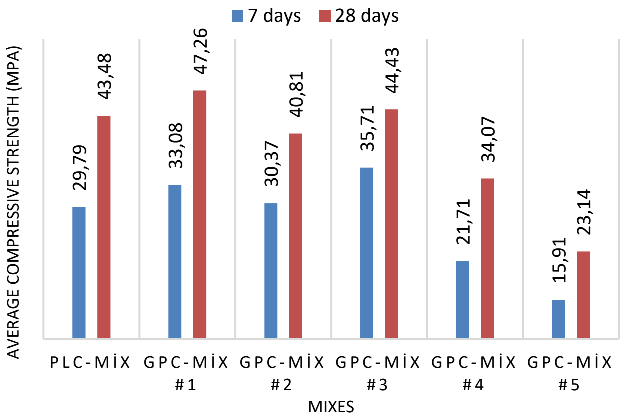

Fig. 8 7 and 28 days average compressive strength of sample mixes

IV. Conclusion

This study investigated the compressive strength of GPC incorporated with different proportions of GGBFS and RHA for structural applications. Experimental methods were adopted to generate the maximum compressive strength of the test samples, and the GPC results were compared with PLC. By the aim and objectives of this study, the following conclusions are made:

1) Replacing PLC with GGBFS and RHA produced stronger concrete after 7 and 28 days of curing. The highest compressive strength obtained from the GPC mixes is 19.87% and 2.18% higher than PLC after 7 and 28 days, respectively.
2) The optimal cementitious materials ratio was demonstrated in GPC-Mix #3 containing 50% GGBFS – 50% RHA with the highest compressive strength of 44.43 MPa. This is because the RHA particles were closely packed, possessed high fineness and increased pozzolanic reactivity in the geopolymerization process.
3) RHA is a possible binder as it increased the strength of the GPC up to 50% replacement in corporation with GGBFS.
4) RHA-blended GPC mixes exhibited low workability because of their particles' large surface area and porous structure.
5) Both the GPC and PLC concrete samples were normal-weight concrete as their densities were within 2200- 2600 kg/m^3 at 28 days of curing, showing slag-based GPC does not affect concrete's density.
6) Using RHA-GGBFS not only produces concrete with enhanced strength but also eradicates the carbon dioxide emission associated with PLC production in the construction industry.

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