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Influence of Different Activated Pozzolans on Compressive Strength of Portland Cement-Activated Pozzolanic Concrete

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Compressive strength, Activated metakaolin, Hybrid concrete, Activated Rice Husk ash, Robotic

Abstract

This study was aimed at evaluating the effects of partial replacement of ordinary Portland cement (OPC) with activated pozzolans (AP) on the compressive strength of concrete produced by hybridizing OPC and AP as the binder. The pozzolans employed were Metakaolin (MK), Rice husk ash (RHA) and Palm oil fuel ash (POFA). The activated pozzolanic binders were produced by activating MK, RHA and POFA with a combination of sodium hydroxide and sodium silicate alkaline solution, which was then used to substitute OPC at 10%, 20% and 30% levels. Concrete with 100% OPC served as the control for all specimens. A mix ratio of 1:2:4 binder to fine aggregate to coarse aggregate by weight was used. Concrete specimens were cured in water for 7, 28, 56 and 91 days. Maximum compressive strength of 23.6 N/mm^2 obtained was for the control specimen at 91 days. The maximum strength obtained for any OPC-AP hybrid concrete was 16.6 N/mm^2 at 10% activated RHA (ARHA), representing 70.3% of the compressive strength of the control and a standard deviation of 4.32 N/mm^2. Also, the best results obtained for OPC-AMK and OPC-APOFA were 15.5 N/mm^2 and 13.7 N/mm^2 respectively representing 65.7% and 58.1% of the control. These values were also obtained at 10% OPC replacement. From the results, the optimum replacement of OPC by any of the activated pozzolan was 10%, further replacement only resulted in loss of strength which is not justifiable. Below 10% replacement, ARHA was better than AMK and APOFA; beyond 10%, AMK was a better binder than ARHA and APOFA when hybridized with OPC due to reactivity and micro-structure differences.

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Introduction

Alkali-activated pozzolan (AP)/ geopolymer are environmental-friendly binders that are produced when a strong alkaline solution such as the combination of NaOH and $NaSiO_3$ is mixed with silica-rich precursors like mata-kaolin and rice husk ash. The use of alkali-activated pozzolan offers the opportunity of a circular economy whereby OPC which is the most carbon-intensive component of concrete is replaced fully or partially (Ahmad, 2022). Since Portland cement (OPC) is typically the most expensive constituent of concrete, replacement of a part of it with a pozzolan offers improved concrete affordability, particularly for developing countries Singh (2018). *When compared to Portland cement, activated pozzolan offers a saving of up to 40% in energy and a 70% decrease in carbon emission (Mclellan et al., 2011)*. Rice husk ash (RHA) can be used to create sustainable and durable activated geopolymer concrete which has the potential of replacing conventional concrete in structural utilization (Parveen, *et al.*, 2021). Combining pozzolans in specific ratios in geopolymer concrete production was established to be viable as an environmentally friendly non-load-bearing masonry alternative (Sukmak *et al.*, 2019 and Detphan *et al.*, 2021)

An AP/ OPC hybrid is an aggregation of alkali-activated pozzolan and OPC, in a bid to create a binderl that combines the beneficial attributes of OPC with the advantageous properties of alkali-activated pozzolan (Marczyk *et al.*, 2021). The rationale for hybridizing OPC and AP was to utilize the initial heat released by the hydration of OPC for heat-curing of the activated pozzolan. Hybridizing OPC and AP helped in producing concrete not requiring thermal curing and yet without loss in compressive strength (Rivera *et al.*, 2014). Hybridizing AP with OPC has been found to reduce the setting time, decrease porosity and greatly increase the compressive strength of geopolymer concrete (Olivia, 2024). The development of a one-part activated pozzolan aims to promote the large-scale application of geopolymers in the construction

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industry. For most one-part mixes, aluminosilicate precursors were blended with solid activators and reactions began once the water was added to the solid mix, when cured under the ambient condition, the resulting binders have improved compressive strength and increased early age strength resulting from the rapid reaction of the OPC with alkali activators (Askarian, 2018). Kumar *et al.*, (2019) confirmed from an experimental study that hybridizing 60 % OPC and 40 % geo-cement is capable of producing a concrete with strength up to 40 MPa. A composite binder, capable of producing compressive strength nearly the same as OPC can be obtained by mixing OPC with 10 % to 20% of highly siliceous material such as Diatomaceous earth powder (Hasan *et al.*, 2021). However, 10% replacement of OPC with activated pozzolan (activated meta-kaolin) was the optimum obtained by Egwuonwu *et al.*, (2019). This study was purposed to determine the effects of partially replacing ordinary Portland cement (OPC) with activated pozzolans (AP) on the compressive strength of concrete produced by hybridizing OPC and those activated pozzolans as binder using water curing.

Materials and Methods

Cement

Ordinary Portland cement comprised of tri-calcium silicate (C_3S), di-calcium silicate (C_2S), tri-calcium aluminate (C_3A), and tetra-calcium aluminoferrite (C_4AF). Dangote cement brand of ordinary Portland cement manufactured in conformity to EN 197 – 1:2000 (2000) was utilized for this research. The grade of the cement was 32.5R. This served as the main binder for all the specimens.

Precursors (Pozzolans)

Calcinated kaolin, rice husk, and palm oil chaffs were used as the precursors for the activated pozzolans. Electric furnace of the Departments of Mechanical Engineering and Industrial Chemistry, Federal University of Technology, Akure were used to create the metakaolin (Figure 1a). A temperature of 650 °C and a 90-minute heat soaking period were used for the calcination. The composition of the primary oxides and a few trace elements in the Mk is displayed in Table 1. The rice husk was sourced from rice mills in Ekiti and Ondo States of Nigeria. It was prepared using an electric furnace in the Department of Mechanical Engineering, Federal University of Technology, Akure. The heat was maintained inside the furnace at a temperature of 600°C (Zaffar *et al., 2022*). The ash was left inside the furnace to cool down before collecting and sieving it to the required fineness. Chemical analysis of the processed RHA was also carried out. Table 1 shows the composition of the main oxides as well as some trace elements in the RHA (Figure 1b). Palm oil chaffs were collected from palm oil mills situated in Akure and Okitipupa in Ondo State. It consisted of palm kernel shells, empty fruit bunches and fruit fibers in the dry state. The palm wastes were burnt at a temperature of 600°C (Zaffar *et al., 2022*). The burnt ash was then crushed and sieved. The composition of the oxides as well as some trace elements in the POFA is shown in Table 1. The sum of SiO_2, Fe_2O_3, and Al_2O_3 in the three pozzolans was more than 70% which confirms their suitability as natural pozzolans as per ASTM C618-12a (2014). The specific gravity for the meta-kaolin was 2.60 with a median particle size of 0.212 mm.

Table 1. Oxides Composition of Metakaolin, Rice Husk Ash, Palm Oil Fuel Ash

Oxide	RHA Oxide %	MK Oxide %	POFA Oxide %
MgO	1.5292	8.9283	5.03
Al_2O_3	5.3438	5.4938	7.65
SiO_2	64.838	73.403	62.19
P_2O_5	4.8697	0.4481	0
SO_3	2.1507	0.8454	0
K_2O	4.9286	0	2.720
CaO	3.7458	0.3914	1.263
$MnO_{(2)}$	0.1613	0	2.98
Fe_2O_3	2.7798	8.3111	5.39
CuO	0.0707	0.0218	4.75
SO_3	2.1507	0.8454	0
K_2O	4.9286	0	2.720
CaO	3.7458	0.3914	1.263
$MnO_{(2)}$	0.1613	0	2.98
Fe_2O_3	2.7798	8.3111	5.39
CuO	0.0707	0.0218	4.75

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ZnO	0.3759	0.0433	2.969
CdO	0	0	4.58
SnO_2	4.4262	0.9004	0
Sb_2O_3	3.6441	0.8910	0
ZnO	0.3759	0.0433	2.969
CdO	0	0	4.58
SnO_2	4.4262	0.9004	0
Sb_2O_3	3.6441	0.8910	0

Figure 1a Metakaolin

Figure 1b Rice husk ash

Alkaline activator: For this work, an alkaline activator consisting of 16M sodium hydroxide solution (NaOH) and sodium silicate (Na_2SiO_3) was employed. The sodium silicate and sodium hydroxide were obtained in liquid form from African Fertilizer and Chemicals, Agbara, in Ogun State, Nigeria. The technical details of the alkaline activators are shown in Tables 2 and 3

.Table 2. Properties of Liquid Sodium Silicate Alkaline

Parameters	Result
Specific Gravity	1.56
% Soda Content (%Na_2O)	15.34
% Silica Content (%SiO_2)	30.70
Wt. Ratio (Na_2O:SiO_2)	1 : 2
% Total Solids	46.04
PH	11.9
Viscosity	1100CP

Table 3. Properties of Caustic Liquid Soda (NaOH)(16M)

Specification	Result
Specific Gravity	1.50
Appearance	Colourless
Sodium Hydroxide (%)	48.24
Sodium Oxide	37.38

Aggregates: The coarse and fine aggregates used were from a quarry in Akure, Ondo State, Nigeria. The coarse aggregate was crushed granite ranging in size from 4.75mm to 19 mm. The sieve analysis and specific gravity were obtained in accordance with BS 12620:2002 (2022) standard. Likewise, natural fine sand that had been graded to a minimum particle size of 0.150 mm and passed a 4.75 mm screen was used as fine aggregate. The sand was graded in conformity to BS 12620:2002 (2022). Figure 2 and Table 4 display the grading curve for the sand and the specific gravity for the aggregates.

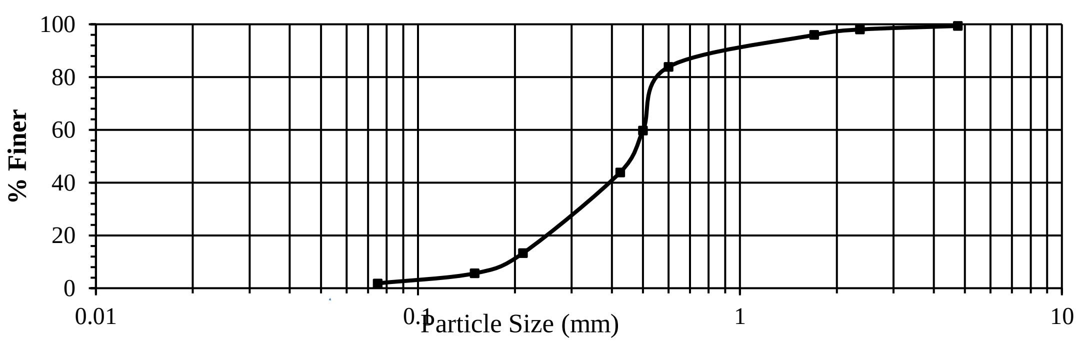

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Figure 2: Particle Size Distribution Curve for Sand

Table 4 Specific gravity of the aggregates

Specification	Result
Sand	2.61
Granite	2.73

Water: The minimum requisite considered for water used in the production of concrete is that it is drinkable, or that it be clean and free from conditions harmful to concrete. Potable water conforming to BS EN 1008:2002 (2002) from the Federal University of Technology, Akure was used for the research work.

Mix Design: For the control and OPC-AP hybrid concrete specimens, a mix ratio of 1:2:4 (binder: fine aggregate: coarse aggregate) adhering to BS5328-2:1997 (1997) was used. From Table 5, it was shown that the control mix was without AP, while the other mixtures contained AMK ARHA and APOFA in varying amounts. Materials were mixed and batched according to weight in kilograms (kg). Activated pozzolans were used to replace OPC at 10%, 20%, and 30% levels. 204 concrete cube specimens were cast.

Table 4 Mix proportions of OPC- Activated Pozzolan concrete (kg/m^3)

Cube ID*	OPC	Mk	Rha	Pofa	Coarse Agg.	Sand	Activator	Total Water
Control	326.0	0	0	0	1304	652.0	0	238
M1A	293.4	32.6	0	0	1304	652.0	14.67	239.4
M2A	260.8	65.2	0	0	1304	652.0	29.34	248.7
M3A	228.2	97.8	0	0	1304	652.0	44.01	251.1
R1A	293.4	0	32.6	0	1304	652.0	14.67	239.4
R2A	260.8	0	65.2	0	1304	652.0	29.34	248.7
R3A	228.2	0	97.8	0	1304	652.0	44.01	251.1
P1A	293.4	0	0	32.6	1304	652.0	14.67	239.4
P2A	260.8	0	0	65.2	1304	652.0	29.34	248.7
P3A	228.2	0	0	97.8	1304	652.0	44.01	251.1

* C = concrete with 100% OPC, M1A = concrete with 10% AMK R2A = concrete with 20% ARHA, P3A = concrete with 30%, APOFA

Alkaline liquid preparation*:* The alkaline solution was the addition of sodium hydroxide (SH) and sodium silicate (SS). After adding the sodium silicate solution to the sodium hydroxide solution, the mixture was thoroughly stirred for five minutes to produce a homogeneous solution. Based on findings from the literature (Faluyi *et al*., 2022; Hardjito and Rangan, 2005 and Aldin *et al*., 2017) an SS/SH mixing ratio of 2.5:1 (Na_2SiO_3): (NaOH) was employed. The resultant alkaline solution was allowed to cool to room temperature before use.

Specimen Preparation: The binders (OPC and pozzolans) were combined with the aggregates in a 1:2:4 ratio (binder: fine aggregate: coarse aggregate). The ratio of the alkaline liquid to each pozzolan was 0.45. Water was added to the alkaline activator solution based on a predetermined water-to-binder ratio. Then, the alkaline activator solution was added to the dry mix (aggregate plus binder) and mixed thoroughly to the requisite consistency to form the fresh OPC/Activated pozzolan hybrid concrete. Finally, the freshly mixed concrete was poured into 150mm x 150mm x 150 mm ready-made molds in three layers and compacted using a tampering rod. For the mix variations, this procedure was repeated. The samples were demolded after 24 hours and stored in a curing tank for 7, 28, 56 and 91 days

Compressive strength test: The 150x150x150 mm concrete specimens were subjected to a compressive strength test following BS EN 196-1:2005. The strength was calculated as the mean value from three specimens following the applicable criteria. The test equipment, depicted in Figure 3, exacts a continuous pressure on the specimen through a top plate connected to a hydraulic ram. The reading was

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automatically taken at the point of failure. Equation (1) was used to get the compressive strength.

$$\text{Compressive Strength (MPa)} = \frac{\text{Maximum Load (N)}}{\text{Cross-sectional Area (mm}^2\text{)}} \quad (1)$$

Figure 3 Compression testing of concrete specimens

Results and Discussion: The partial replacement of OPC with AMK ARHA and APOFA no doubt has an evidential effect on the 7th, 28th, 56th and 91st day compressive strength of the resulting concrete. Figure 4 shows the comparison between the 100% OPC (control) concrete and 10% replacement by AMK ARHA and APOFA. The difference in strength is quite significant. At 91 days while OPC concrete reached 23.6 N/mm^2, OPC-ARHA concrete was the closest at 16.6 N/mm^2. It can then be concluded that at 10% replacement of OPC by Activated pozzolans, OPC concrete is 34.32% better than OPC-AMK concrete, 29.7% better than OPC-ARHA concrete and 42% better than OPC-APOFA concrete at 91 days.

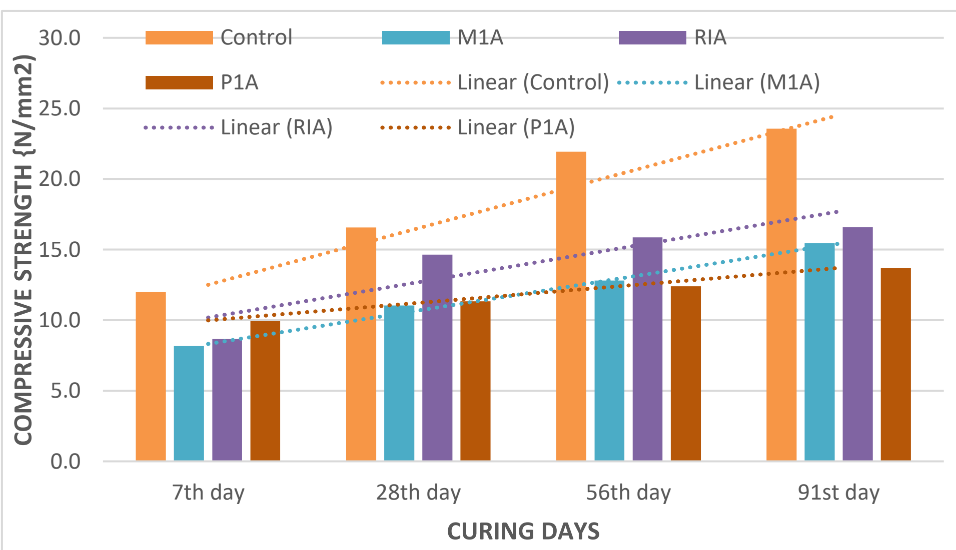

Figure 4 Comparison Between Control and 10% Replacement of OPC by AMK ARHA and APOFA

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At 20% replacement of OPC with AMK ARHA and APOFA, the gap in compressive strength between the control and the OPC-AP concretes became wider as seen in Figure 5. The maximum result obtained for the OPC-AP concretes was for OPC-AMK at 11.7 N/mm^2 at 91 days of curing. It can be concluded that at 20% replacement of OPC by activated pozzolan, the strength of OPC-Mk concrete was only 50% of OPC concrete, OPC-ARHA concrete was 45% that of OPC concrete and OPC/POFA was 42% of OPC concrete at 91 days.

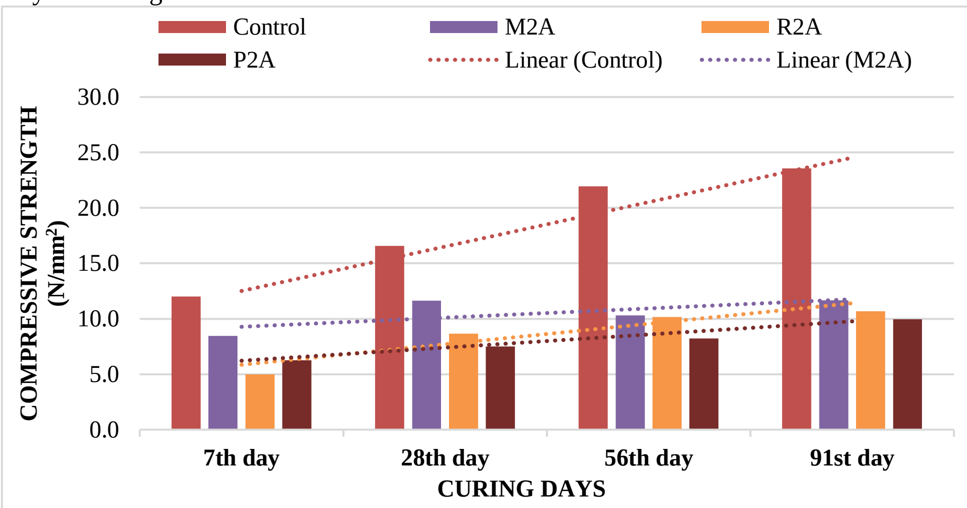

Figure 5 Comparison Between Control and 10% Replacement of OPC by AMK ARHA and APOFA

At 30% replacement of OPC with AMK ARHA and APOFA, the compressive strength of the control was way ahead of any of the OPC-AP concretes. The maximum result obtained for any of the OPC-AP concrete was 10.7 N/mm^2 which was for OPC-AMK concrete at 91 days. With a strength of 23.6 N/mm^2, the compressive strength of OPC concrete was more than twice the best of any of the OPC-AP concrete at 30% activated pozzolanic content. This is shown in Figure 6.

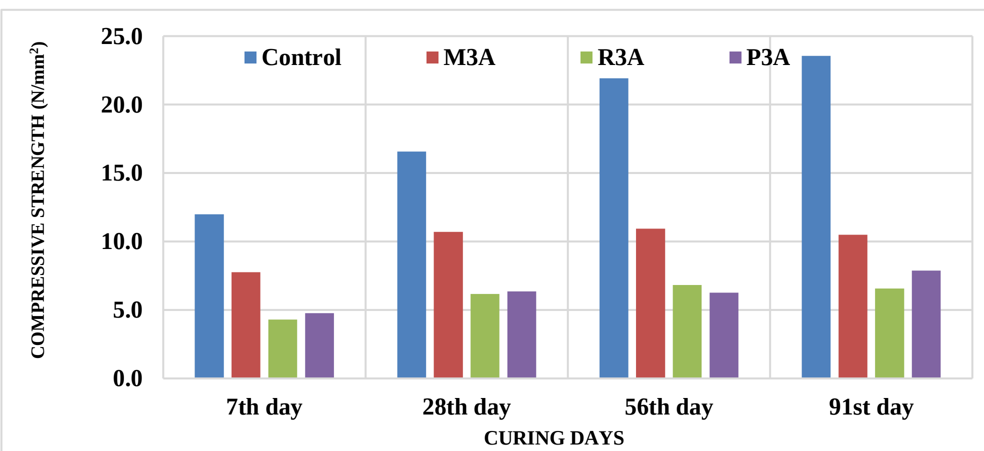

Figure 6 Comparison Between Control And 30% Replacement of OPC by AMK ARHA and APOFA

Expectedly, all the concrete specimens gained strength as curing days increased from 7 through 91 days. It was evident that the concrete specimens gained strength more rapidly in the earlier days before slowing down at the 56th and 91st days

It was shown in Figure 7 that with an increasing replacement of OPC with AMK the compressive strength of the OPC-AMK concrete reduced. The maximum strength obtained with 10% AMK was 15.5 N/mm^2 why AMK at 20% was 11.7 N/mm^2 and AMK at 30% was 10.5 N/mm^2; all of which happened to be

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the strength on the 91st day . An average of 4.4 N/mm^2 loss in compressive strength for every 10% increase in AMK and decrease in OPC. However, the difference in strength loss between the successive replacements was not constant. It was more pronounced at 10% replacement (8.1 N/mm^2), reduced to 3.8 N/mm^2 at 20% AMK and to 1.2 N/mm^2 at 30% replacement. It should also be noted in Figure 8 that while the control still gained some considerable strength after the 28th day, OPC-AMK concrete gain was insignificant after the 28th day except at the 10% replacement.

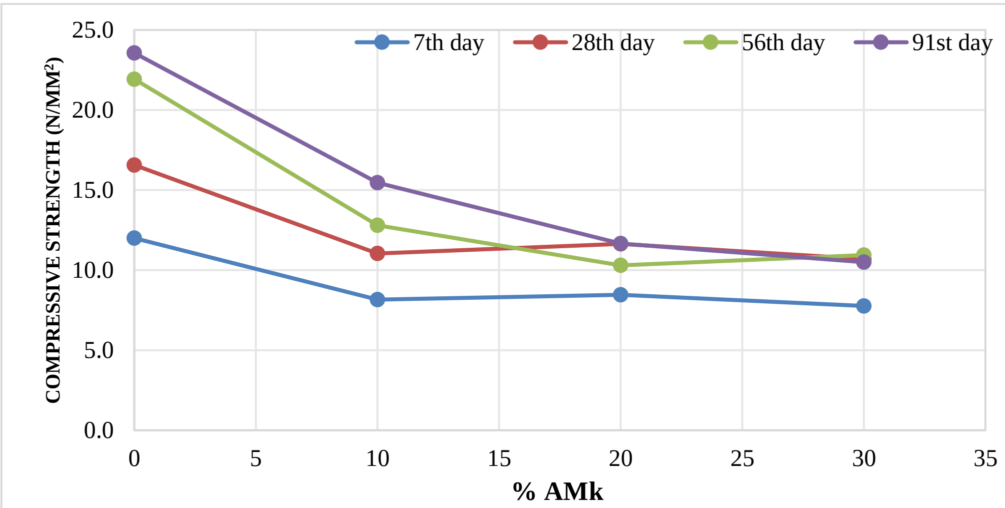

Figure 7 Effect of AMK Variation on Compressive Strength of OPC-AMK Concrete

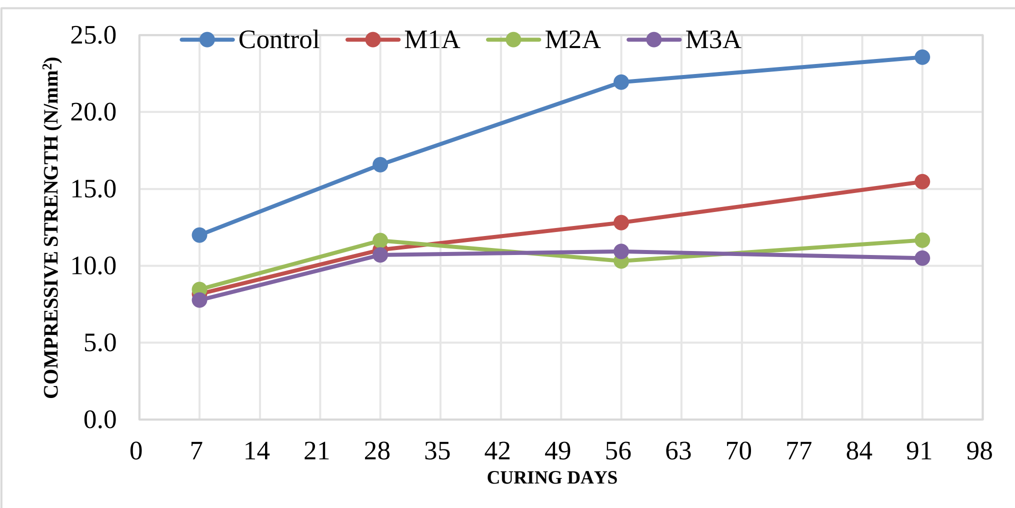

Figure 8 Effect of curing duration on compressive strength of various OPC-AMK Concrete

Replacing OPC with ARHA in concrete caused a reduction of the compressive strength of the resulting OPC-ARHA concrete. From Figure 9, it can be deduced that with increasing ARHA in the concrete, there was a decrease in the strength of the concrete. The highest strength obtained was 16.6 N/mm^2 at 10% replacement. The strength reduced to 10.7 N/mm^2 for 20% replacement and 6.6 N/mm^2 for 30% replacement at 91 days. An average decrease of 5.7 N/mm^2 for every 10% increase in ARHA from 10% through 30% substitution. However, it is essential to note that a difference of 7 N/mm^2 was observed between the

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control (100% OPC) and 10% RHA replacement of OPC in the OPC-ARHA concrete after 91 days of curing as seen in Figure 10. The difference in strength loss between the successive replacements was not constant but more regular than that of AMK discussed earlier. The loss was 7.0 N/mm^2 for the first 10% replacement, 5.9 N/mm^2 for the next 10%, and 4.1 N/mm^2 between 20% and 30% replacement. According to Buyondo *et al.*, (2020) RHA has high specific surface area due to its fineness, this caused higher demand for water thus creating pores when dried up and weakening the matrix. A further explanation for observed strength reduction could be attributed to the water curing regime which particularly did not favour ARHA.

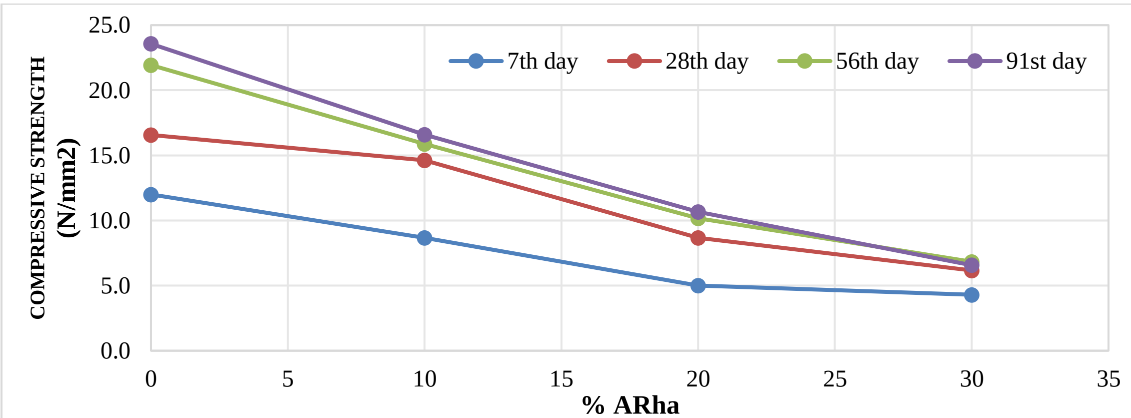

Figure 9 Effect of ARHA Variation on Compressive Strength of OPC-ARHA Concrete

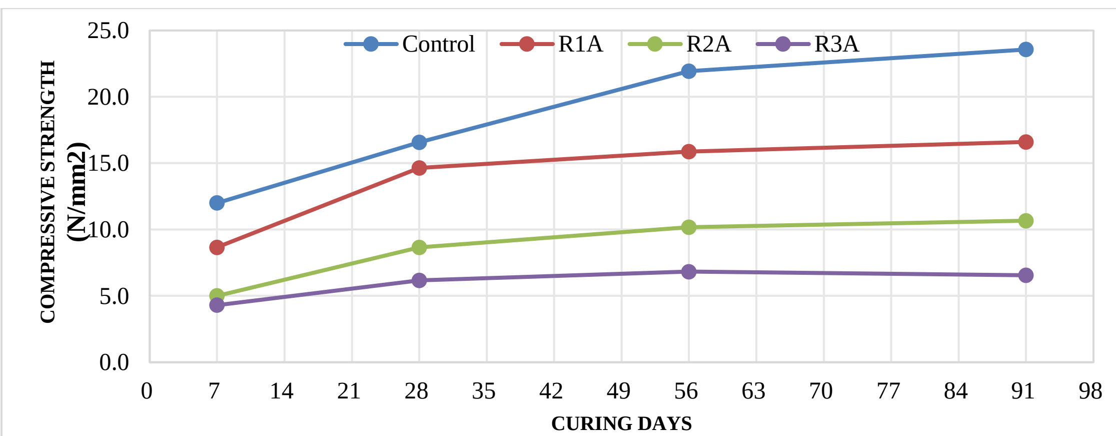

Figure 10 Effect of curing duration on compressive strength of various OPC-ARHA Concrete

A steady increase in the content of activated Pofa in OPC-APOFA concrete resulted in a rapid decline in the compressive strength. From 10% through 20% to 30%, APOFA was used to replace OPC and the corresponding compressive strength was 13.7 N/mm^2, 10 N/mm^2 and 7.9 N/mm^2 respectively. The highest strength obtained was just 58.1% of the control at a mere 10% replacement as shown in Figures 11 and 12. At 30% replacement, OPC-APOFA concrete was just 33.4% of the compressive strength of the control concrete. The strength of APOFA was less than the compressive strength of OPC

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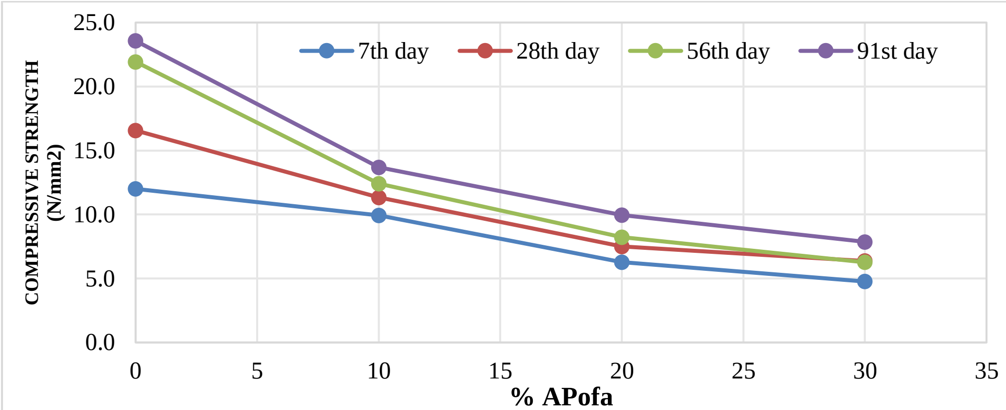

Figure 11 Effect of APOFA Variation on Compressive Strength of OPC-APOFA Concrete

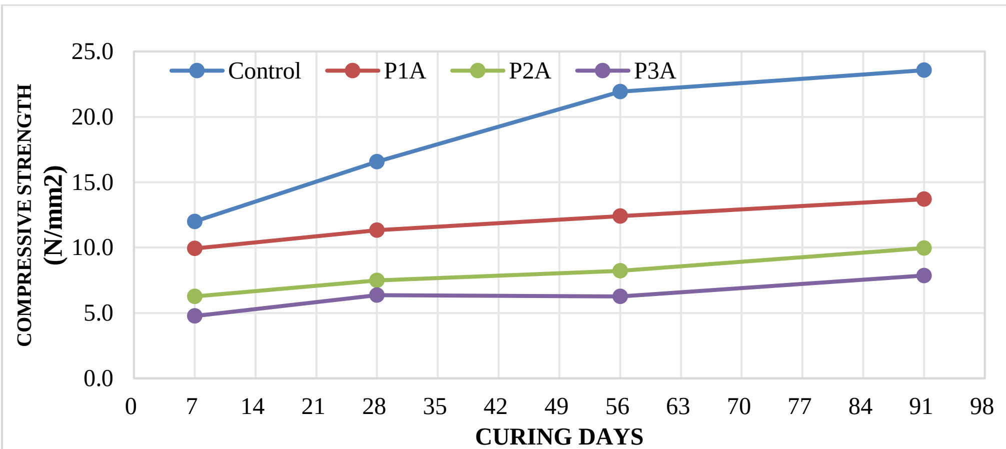

Figure 12 Effect of curing duration on compressive strength of various OPC-APOFA Concrete

Overall, from Figure 13, the performance of each of the activated pozzolan can be seen. At 10% replacement, ARHA was better than AMK and APOFA while AMK was better than ARHA and APOFA at 20% and 30%. At a mere 20% replacement, OPC-AMK concrete was about half of the compressive strength of OPC concrete, while OPC-ARHA and OPC-APOFA were far below the half mark of OPC concrete.

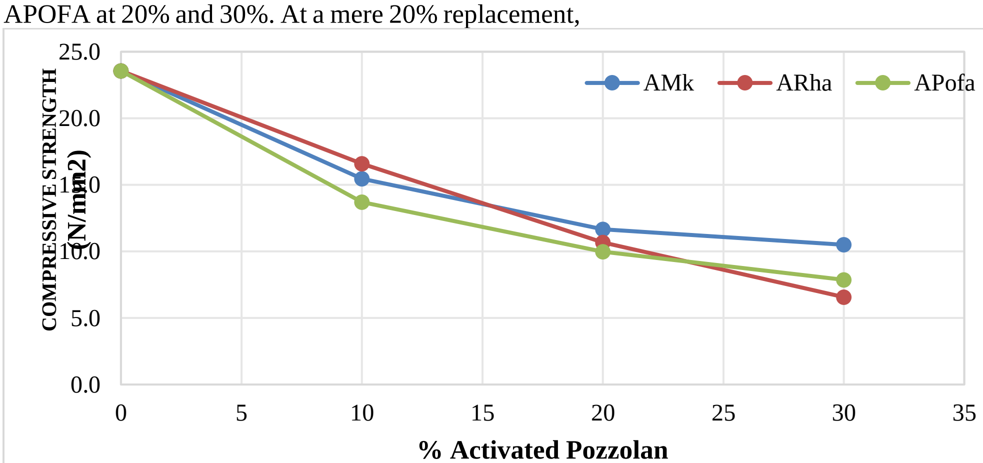

Figure 13 Comparison of the performance of each activated pozzolan at 0% to 30% replacement of OPC

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The influence (feature importance) of each of the activated pozzolan on the compressive strength of the resulting concrete is shown in Figure 14 (beeswarm plot) and Figure 15 (bar plot) using SHapley Additive exPlanations (SHAP) values. From Figure 15 it could be observed that AMK has the least influence on the compressive strength among the activated pozzolans, while APOFA was the most influential albeit negatively. It can be seen in Figure 14 that higher percentages/ values (in red) of OPC caused an increase in the compressive strength of the concrete, likewise, higher values of AMK add little to the compressive strength. Higher quantities of both APOFA and ARHA are detrimental to the strength of the concrete. It was clear that none of AMK ARHA and APOFA could match the strength capability of OPC when water curing was employed. This is supported by findings from Muracchioli *et al.*, (2023) that low temperature curing damage activated metakaolin concrete.

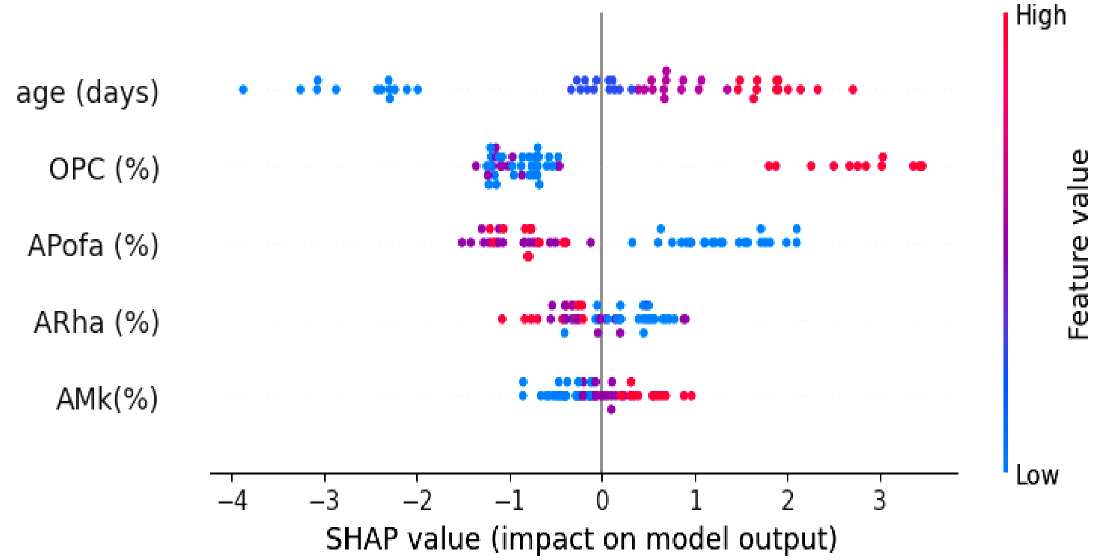

Figure 14 Swarm plot of the variables on the compressive strength of the OPC-AP concrete

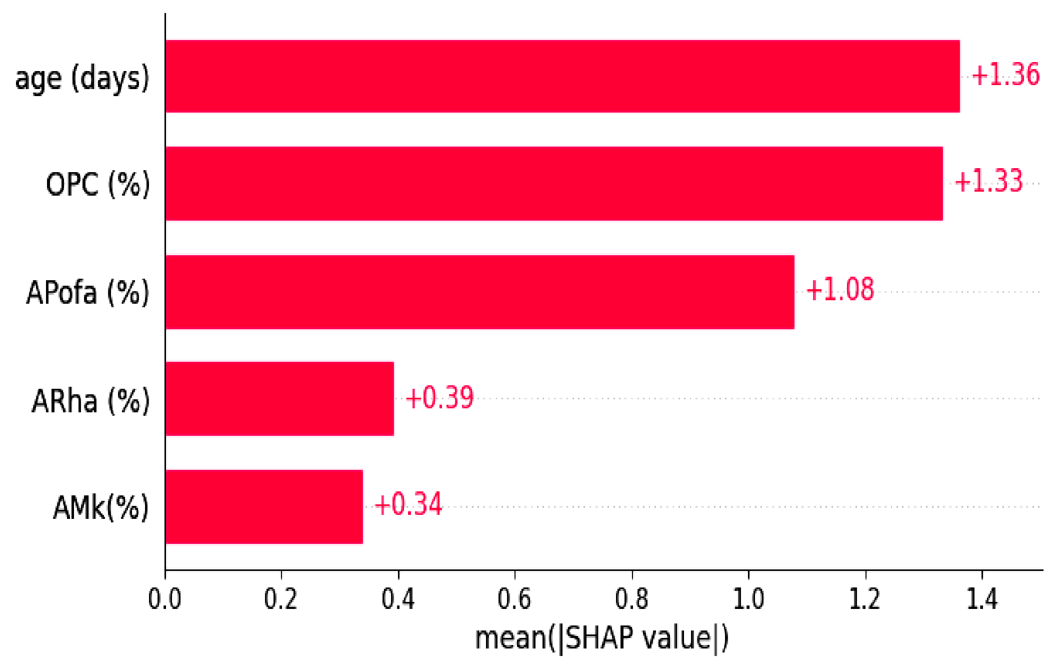

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Figure 15 Influence of each activated pozzolan on compressive strength of the concrete

To verify the reliability of the results, a plot of the standard deviations for the various replacement ratio is shown in Figure 16. It can be shown that the standard deviation increased as the percentage of AP increased. The assumed standard deviation of a plain concrete is expected to range between 3.5 N/mm^2 to 6 N/mm^2 according to IS 456 (2000). It was only when AP was limited to 10% that this was achieved. The increased standard deviation resulted from wide gap between the compressive strength of the control and the OPC-AP concrete as AP content increased. This is an indication that the result is more reliable at lower AP inclusion than at higher levels.

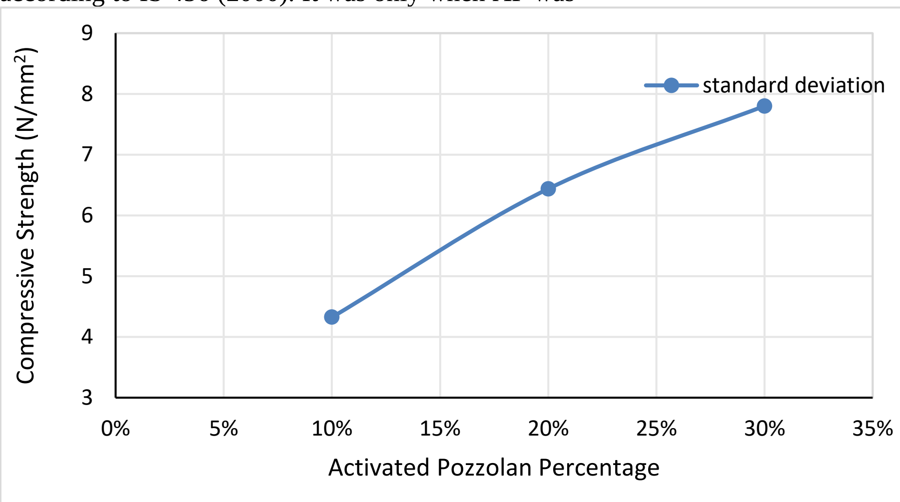

Figure 16: Compressive strength standard deviation

Conclusions

OPC-AP hybrid concrete. However, this research has shown that the AP component of the binder will require more than the hydration heat released by OPC to cure properly, judging by the compressive strength results obtained. Further work is being suggested to find a compromise curing regime such as steam for OPC-AP hybrid concrete. The optimum replacement of OPC by any of the activated pozzolan in OPC-AP hybrid concrete was 10%, further replacement resulted in a significant loss of strength which is not justifiable. The strength of AP could not be maximized when cured in water as against using heat. This was also observed by Castillo *et al.*, (2021) and Nurruddin *et al.*, (2018) that it will take more than ambient temperature to cure AP concrete. Increasing the percentage of AP in the concrete meant reducing the quantity of OPC, a stronger binder when cured in water. Thus increasing AP in the concrete beyond 10% led to overall reduction of strength. Below 10%, ARHA is better than AMK and APOFA. AMK can be adjudged the best of the three activated pozzolan beyond 10% replacement because strength loss is not as rapid in AMK compared to ARHA and APOFA as the replacement level increases. In addition, AMK exhibited better and denser microstructure during geopolymerization compared to ARHA and APOFA (Jindal *et al.*, 2021), owing to its higher reactivity and hydraulic characteristic.

Conflicts of Interest

The authors declare that they have no conflicts of interest to report regarding the present study.

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