Effect of Different Sulphate Types and Concentrations on Compressive Strength of Periwinkle Shell Ash Blended Cement Concrete

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Abstract-- **The study investigated the compressive strength performance of periwinkle shell ash (PSA) blended cement concrete exposed to sulphate environments. Periwinkle shells were obtained from Ikot Ekpene, Akwa Ibom state, Nigeria; and calcined in a furnace to temperature of 800^{0}C at zero soaking time.**

Concrete mix of design characteristic strength of 25N/mm^2 was adopted as the control. The cement component replaced with PSA at five levels of 0%, 10%, 20%, 30% and 40% were cast, and on hardened exposed to varying concentration of sulphates of magnesium, sodium and calcium at four levels of 0%, 1%, 3% and 5% each for three exposure periods of 62, 92 and 152 days after complete immersion in water for 28 days. Using three replicates in all the tests, a total of 630 cubical (150mm) specimens were cast and tested.

The results revealed that compressive strength increased with increase in curing age but decreased as the PSA contents increased. The design compressive strength was attained with 10% PSA content at the age of 28 days. The compressive strength reduction in sulphate solutions was noted to increase significantly ($p = 0.005$, $R^2 = 0.995$) with increase exposure period and concentration, with the most severe caused by magnesium sulphate and the least by calcium sulphate. The least reduction in compressive strength was experienced with 10% PSA blended cement concrete.

Based on the test results the study concluded that 10% PSA content is adequate as supplementary cementitious material for structural concrete to be placed in an aggressive sulphate environment.

Index Term-- **Compressive strength, Concentration, Periwinkle shell Ash, Sulphates, Blended cement concrete.**

1. INTRODUCTION

The compressive strength of concrete is considered one of the most important properties in the hardened state, and the design of concrete structures is based primarily to resist compressive stresses. However, the compressive strength performance and the service life of concrete may be inhibited by its exposure condition. The ability of concrete to resist weathering action, chemical attack, abrasion, or any other process of deterioration is called durability [1]. Inadequate durability manifests itself by deterioration, which can be caused by external or internal factors within the concrete. As stated by [2], the external factors include chemical attack or mechanical damage caused by impact, abrasion, erosion or cavitations; while the internal factors result from chemical reactions involving the concrete constituents.

To mitigate the effect of chemical attack on concrete, mineral admixtures have been used to partially replace cement in concrete [3], [4]. Commonly used mineral admixtures have been fly ash, blast furnace slag, silica fume and rice husk ash. Recently the use of periwinkle shell ash (PSA) as cementitious supplementary material has been reported by [5], [6] and [7] in concrete and sandcrete block production. The use of PSA on concrete has been on its effect on the compressive strength up to hydration period of 28 days; this study rather, examined the compressive strength performance of PSA blended cement concrete in an aggressive sulphate environment.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Ordinary Portland cement (OPC) produced in conformity to [8] was used. Periwinkle shell ash (PSA) was processed from the calcination of periwinkle shells collected from one of the dumpsites in Ikot Ekpene, Akwa Ibom state of Nigeria, to a temperature of 800 ^{0}C at zero soaking time. The ash was ground and sieved with 45µm size. The fine aggregate was that of river-bed sand passing 4.75mm sieve and falls within zone 2 as shown in Figure 1; while the coarse aggregate was crushed granite of maximum size 20mm. The properties of the materials are presented in Tables 1 and 2.

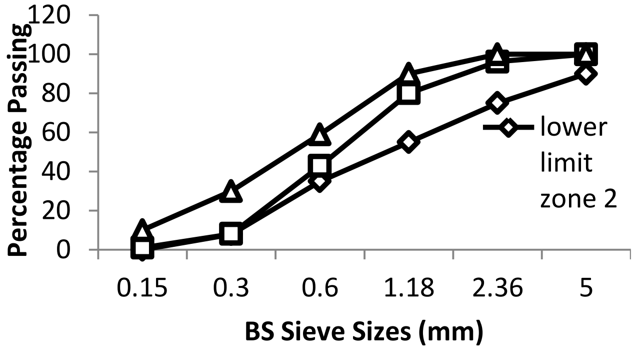

Fig. 1. Zone two grading curve of fine aggregate

TABLE I
Physical Properties of Materials

Properties	cement	PSA	Fine aggregate	Coarse aggregate
Fineness Modulus	-	-	2.72	6.39
Coefficient of Uniformity	-	-	2.50	2.40
Specific gravity	3.13	2.13	2.65	2.65
Consistency (%)	27.72	-	-	-
Initial setting time (minutes)	98	-	-	-
Final setting time (minutes)	201	-	-	-
Fineness (% retained on 45μm sieve)	32.00	21.00	-	-
Soundness (mm)	0.08	1.00	-	-
Water requirement (% of control)	-	104	-	-
Moisture content (%)	-	1.5	-	-
Pozzolanic activity with Portland cement (% of control): 7 days	-	78.17	-	-
28 days	-	79.12	-	-

TABLE II
Chemical Properties of Cementitious Materials obtained by XRF Analysis

Elemental oxide	OPC	PSA
SiO_2	20.06	33.84
Al_2O_3	5.85	10.20
Fe_2O_3	3.05	6.02
CaO	62.44	40.84
MgO	1.93	0.48
SO_3	2.71	0.26
K_2O	0.97	0.14
Na_2O	0.14	0.24
Mn_2O_3	0.20	0.00
P_2O_5	0.17	0.01
TiO_2	0.28	0.03
LOI	1.09	7.60

2.2 Proportioning and mixing of constituents

The mix proportion was based on mix design of characteristics strength of 25 N/mm^2 at 28 days for normal concrete (i.e. control: 0% PSA and 100% OPC). A water/cement ratio (0.58) requirement of the mix design for the requisite workability (slump: 10-30 mm) was adopted. The cement was blended with different percentages of PSA in the range of 0-40% at intervals of 10% by volume since their specific gravities were significantly different as weight replacement will amount to higher quantity of PSA for the same weight of cement.

The ingredients, that is, cement, PSA, aggregates and water, were manually mixed on an impermeable platform. The blended cement and PSA was spread on already measured sand, and the three ingredients mixed thoroughly before the coarse aggregate and water were added. Mixing was assumed to be completed when a homogenous mix has been obtained.

2.3 Workability Tests on fresh concrete

Slump and compacting factor tests were carried out on the fresh concrete to determine the workability of each mix. The tests were done in accordance with the requirement of [9] for slump test and [10] for compacting factor test.

2.4 Casting and curing of specimens for test

Before casting, the moulds were thoroughly cleaned and coated with mould oil to ensure easy de-moulding and smooth surface finish. The casting was done in accordance with the requirements of [11]**.** The cubes were de-moulded at 24 hours, and immersed in water curing tanks kept at a temperature of 29.0 ± 1.0 ℃ up to 180 days. Some specimens were immersed in three different sulphates, namely magnesium, sodium and calcium sulphates, solutions of various concentrations of 0, 1, 3 and 5% after 28 days of water curing.

2.5 Compressive strength test

The compressive strength test was carried out as specified by [12]. Two sets of 150 mm cubes totalling 630 were used for the determination of compressive strength of PSA blended cement concrete. The first set consists of 90 cubes and used for determining the effect of incorporating PSA on the compressive strength at curing ages of 7, 14, 28, 90, 120 and 180 days; while the second set of 540 cubes were used to assess the effect of different sulphate types and concentrations on the compressive strength of PSA blended cement concrete at 62, 92 and 152 days after water curing for 28 days. The degree of deterioration was evaluated by measuring the reduction in compressive strength by the expression:

$$RCS (\%) = [(Cc-Cs)/Cc] \times 100$$

Where RCS is reduction (or increase) in compressive strength,

Cc is the average compressive strength of three specimens cured in water, and

Cs is the average compressive strength of three specimens exposed to sulphate solution.

3. Results and Discussion

3.1 Workability test on fresh concrete

The results of the slump and compacting factor (CF) as shown in Table 3 indicated that to attain the same low workability level of 10–30mm in the mixes containing PSA with that of conventional concrete (i.e. control), higher water content was required. This is reflected in the gradual increase in the water/binder ratio with a corresponding increase in the amount of water over control as the PSA percentage content increases. This higher water requirement in mixes containing PSA could be attributed to its high fineness which meant a greater specific surface to be wetted and lubricated; this agreed with the earlier finding by [13] which used rice hush ash as partial replacement of cement in concrete.

TABLE III
Slump and Compacting Factor values for PSA Blended Cement Concrete

PSA content (%)	Slump (mm)	Compacting Factor	Actual Water/Binder Ratio	Amount of Water over Control (%)
0	29	0.86	0.58	100.00
(control)	28	0.87	0.59	101.72
10	28	0.85	0.60	103.45
20	26	0.84	0.62	106.29
30	25	0.83	0.64	110.34
40				

It was observed, as shown in figure 2, that there is a direct relationship between the slump values with that of the compacting factor in all the mixes. Decrease in the value of the slump caused a corresponding decrease in the value of the compacting factor with a correlation coefficient of 0.866.

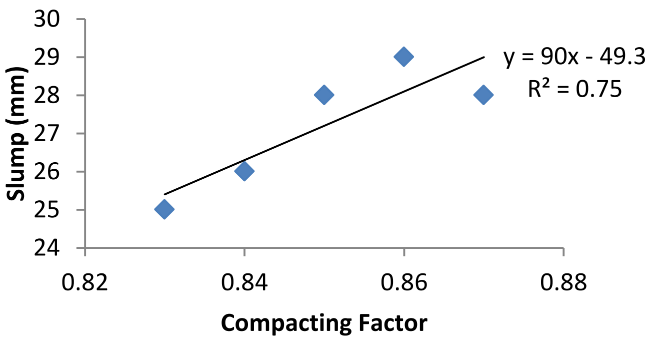

Fig. 2. Relationship between compacting factor and slump of PSA blended Cement concrete

3.2 Compressive strength of specimens cured in water

The mean compressive strength for each replacement level of cement with PSA at 0%, 10%, 20%, 30% and 40% and for different curing ages of 7, 14, 28, 90, 120 and 180 days as well as its percentage attainment of the design strength of 25 N/mm^2 at 28 days are shown in figures 3, 4 and 5. The compressive strength generally increases with curing age and decreases with increased content of PSA. The results at 7 days as presented in figure 5 show that in all the replacement levels the percentage attainment of the design strength range between 77.63% and 65.66% with 0% PSA content (i.e. control) having 77.63% and 40% PSA content having the least value of 65.66%. These values satisfied the requirement of normal concrete strength development which is stipulated to be between 50-66% [14].

At 14 days hydration period, the control mix (i.e. 0% PSA) have exceeded the design strength as its strength increased to 27.11 N/mm^2, representing 108.44% of the design strength; this is closely followed by 10% PSA which had 85.33% of the design strength, while 10%, 20% and 30% PSA replacement had compressive strength of 18.04, 17.01 and 16.30 N/mm^2 representing 72.18, 68.04 and 65.19% of the design strength respectively. The strength development at 14 days satisfied the 60-75% of the design strength as stipulated by [15].

The compressive strength of 0% PSA and 10% PSA content at 28 days hydration period were 28 N/mm^2 and 25.56 N/mm^2 respectively which met the desired design strength of 25 N/mm^2, while that of 20%, 30% and 40% PSA content were 24.15 N/mm^2, 20.71 N/mm^2 and 15.91 N/mm^2 respectively. This reveals that 10% PSA content is adequate because it met the 28-day design strength. These values are comparable with that of [5] which recorded values of 30.69 N/mm^2, 25.78 N/mm^2, 22.67 N/mm^2, 20.00 N/mm^2 and 16.00 N/mm^2 for 0%, 10%, 20%, 30% and 40% PSA content at 28 days, but lower than that of [6] which recorded values of 31.92N/mm^2, 30.19 N/mm^2, 26.08 N/mm^2, 25.17 N/mm^2 and 25.06 N/mm^2 for 0%, 10%, 20%, 30% and 40% PSA content respectively. The strength development for control mix (i.e. 0% PSA) was faster up to 28 days hydration period where as mixes containing PSA was slower; this portray the fact that the pozzolanic reaction depends on the release of calcium hydroxide from cement hydration. With compressive strength of up to 20 N/mm^2 obtained for percentage replacement up to 30%, it shows that PSA can still be used effectively up to 30% replacement level in PSA/OPC cementation mixture for construction of low-rise concrete buildings exposed to fair weather.

The results at 90 days indicated that for all the mixes there is continuous increase in the strength, showing that there is both hydration and pozzolanic reactions particularly with 10% PSA having a higher rate of development than the control. At 120 days, 10% PSA recorded strength of 28.53 N/mm^2 representing an increase of 6.89% of the strength at 90 days, while the control mix recorded strength of 29.92 N/mm^2 which represents an increase of 3.26% of the strength of 90 days. Other mixes had little or no increase in the design strength beyond 90 days. The 20% PSA had strength of 24.89 N/mm^2 at 120 days, representing 99.56% (approximately 100%) of the design strength. It means that where later age strength is required at 120 days hydration period, 20% replacement of cement with PSA is adequate.

A further increase in the rate of strength development was observed with 10% PSA at 180 days as it attained strength of 29.04 N/mm^2 which is not significantly different from the control which had strength of 30.15 N/mm[2]. The continuous increase in the 10% PSA can be attributed to the fact that the quantity of calcium hydroxide liberated from cement hydration is adequate to be consumed by the pozzolanic reaction.

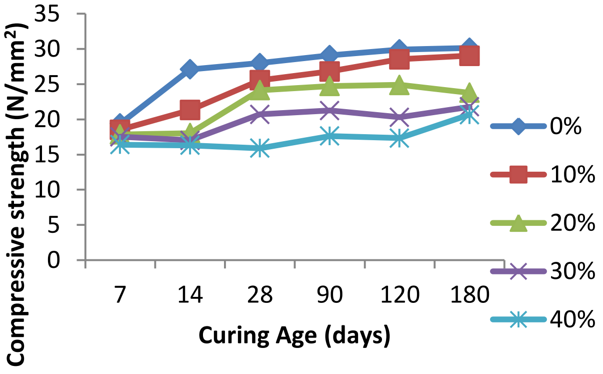

Fig. 3. Variation of compressive strength with different curing ages for varying percentages PSA contents.

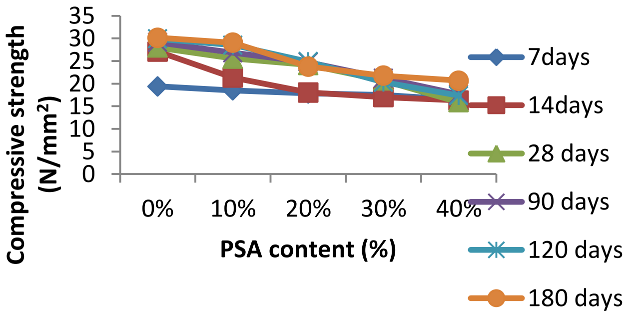

Fig. 4. Variation of compressive strength with varying Percentages of PSA contents for different curing ages.

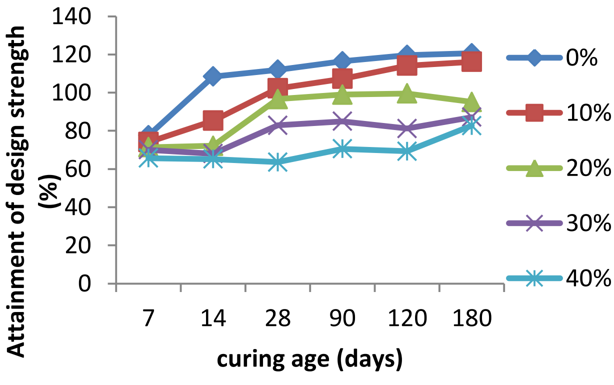

Fig. 5. Percentage attainment of design compressive strength at 28 days for different levels of PSA content.

3.3 Compressive strength of specimens immersed in sulphate solutions

The results of sulphate attack on compressive strength expressed as the strength change (in percentage) based on percentage PSA content for different sulphate types and concentrations is shown in figures 6 to 14.

There was a strength reduction of 1.51% when the concrete specimen of 0% PSA content was exposed to 1% $MgSO_4$ solution at 62days; whereas there was gain in strength of 0.3%, 1.05%, 2.19% and 3.35% by 10% PSA, 20% PSA, 30% PSA and 40% PSA content, respectively. When the specimens were in 1% Na_2SO_4 solution up to 62days, 0% PSA content loss strength by 0.96% while others had strength increased by 4.44%, 4.24%, 5.41% and 6.86% for 10% PSA, 20% PSA, 30% PSA and 40% PSA replacement of cement respectively. In the $CaSO_4$ (1% solution), all specimens had improvement in their compressive strength as they recorded 1.61%, 3.10%, 5.13%, 6.69% and 8.91% for 0% PSA, 10% PSA, 20% PSA, 30% PSA and 40% PSA content, respectively. The highest strength loss of 8.82%, 8.56% and 7.69% were recorded by 0%PSA content in $MgSO_4$, Na_2SO_4 and $CaSO_4$ solution respectively at 92days exposure. The loss in strength with 10% PSA to 40% PSA were between 3.29% and 3.82% for that of $MgSO_4$; Na_2SO_4 and $CaSO_4$ range from 1.38% to 3.15%, and 1.09% to 1.85%, respectively. At 152 days, 0% PSA had strength loss of 9.35%, 9.19% and 8.19% in magnesium, sodium and calcium sulphate solutions, respectively. Values of 2.82%, 0.59%, 2.02% and 2.08% loss in strength were recorded in $MgSO_4$; while 2.82%, 0.93%, 1.01% and 1.26% in Na_2SO_4 and 2.07%, 1.85%, 1.42% and 1.16% in $CaSO_4$ solution were recorded for 10% PSA, 20% PSA, 30% PSA and 40% PSA content, respectively.

In 3% sulphate concentration, 0% PSA and 10% PSA content had loss in strength of 2.23% and 1.38%, while 20% PSA, 30% PSA and 40% PSA had improvement of strength of 0.16%, 1.58% and 3.57% in that order when in $MgSO_4$ solution for 62 days. In Na_2SO_4 solution, only 0% PSA concrete specimens' loss strength by 1.55% while other concrete specimens gained strength ranging from 1.09% to 4.88%. In $CaSO_4$ solution, all the specimens gained strength ranging from 1.03% to 9.42%. At 92 days exposure, loss in strength were recorded by specimens in $MgSO_4$ and Na_2SO_4 solution for all the replacement levels of 0% to 40% PSA, but 0% PSA had the highest reduction of 11.56% and 11.36%, respectively. A gain in strength was recorded with 30% PSA and 40% PSA whereas 0% PSA had strength loss of 8.69% when the specimens were exposed in $CaSO_4$ solution. At 152 days exposure, 0% PSA concrete specimens had the highest lost in strength of 15.46%, 9.68% and 5.67% when exposed in $MgSO_4$, Na_2SO_4 and $CaSO_4$ solution, respectively. The least strength loss was 3.10% in $MgSO_4$ and 1.52% in Na_2SO_4 for 10% PSA specimens. Values of 2.03%, 2.82%, -0.14% and -1.26% were obtained for 10% PSA, 20% PSA, 30% PSA and 40% PSA concrete specimens, respectively, in $CaSO_4$ solution.

Equally, at 62days in a 5% sulphate solution, concrete containing 0% and 10% PSA recorded a strength reduction of 3.26% and 1.42%, while strength gained of 0.16%, 1.11% and 2.59% were recorded with 20%, 30% and

40% PSA content, respectively. In Na_2SO_4, at same exposure period, 0% PSA and 20% PSA had strength loss of 3.44% and 0.12% respectively, while 10% PSA, 30% PSA, and 40% PSA improved their strength by 3.62%, 0.42% and 2.89%, respectively. Calcium sulphate solution had a strength improvement of 1.00%, 2.80%, 1.51% and 8.96% for 0%, 10%, 30% and 40% PSA content, respectively whereas 20% PSA content had a loss of 0.12% in its compressive strength when exposed in 5% $CaSO_4$ solution for 62 days.

At 92 days exposure, all the specimens recorded reduction in their compressive strength irrespective of the type of sulphate. In $MgSO_4$ solution, reduction in compressive strength were 14.87%, 4.59%, 5.26%, 5.96% and 5.08% for 0, 10, 20, 30 and 40% PSA content respectively. Values of 15.34%, 4.21%, 3.29%, 5.12% and 4.27% were obtained for 0, 10, 20, 30 and 40% PSA content, respectively in Na_2SO_4 solution; while in $CaSO_4$ solution, 8.92% to 1.56% were recorded for 0% to 40% PSA content in that order.

With a sulphate concentration of 5% at 152 days exposure, magnesium sulphate exhibited the most severe effect on the concrete compressive strength with compressive strength loss of 19.67%, 3.65%, 4.67%, 4.09% and 4.02% closely followed by sodium sulphate with values of 11.58%, 2.34%, 2.82%, 2.98% and 3.29% for concrete containing 0%, 10%, 20%, 30% and 40% PSA, respectively. Calcium sulphate had the least effect with strength reduction of 7.40%, 2.10%, 2.94%, 2.16% and 2.32% for 0% PSA, 10% PSA, 20% PSA, 30% PSA and 40% PSA content in that order. A minimal strength loss was observed with 10% PSA concrete specimens.

Generally, the strength reduction became much manifested with increase in exposure age particularly at 152 days. Magnesium sulphate had the most deleterious effect on the concrete compressive strength especially on 0% PSA content (i.e. the control) than other concrete specimens with various percentages of PSA replacement levels with cement. The effect of sodium sulphate solution on the concrete specimens was equally deleterious but not as severe as that of the magnesium sulphate solution though more severe than that of calcium sulphate solution. Calcium sulphate solution had the least effect when compared with magnesium and sodium sulphates. Also noticed was a decrease in strength loss as the percentage of PSA content increases from 10% to 40% particularly at 62 and 92 days exposure in all the solutions. Beyond this age, the least loss in compressive strength was experienced with 10% PSA blended cement concrete specimens. It was also noticed that the value of strength loss increases as the concentration of the sulphate increases from 1% to 5% concentration in all the sulphates with the most severe caused by magnesium sulphate and the least by calcium sulphate.

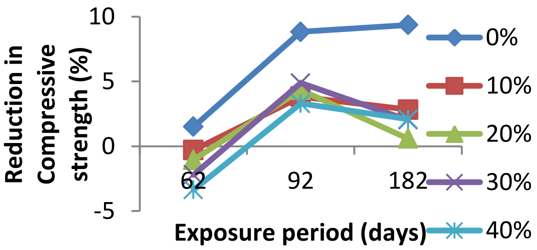

Fig. 6. Reduction in compressive strength of PSA blended cement concrete exposed to 1% $MgSO_4$ solution

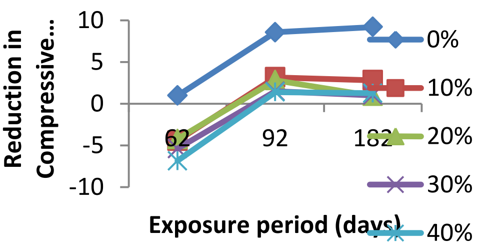

Fig. 7. Reduction in compressive strength of PSA blended cement concrete exposed to 1% Na_2SO_4 solution

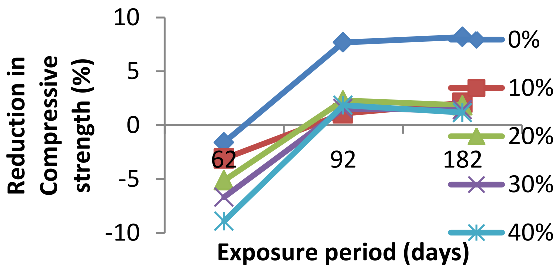

Fig. 8. Reduction in compressive strength of PSA blended cement concrete exposed to 1% $CaSO_4$ solution

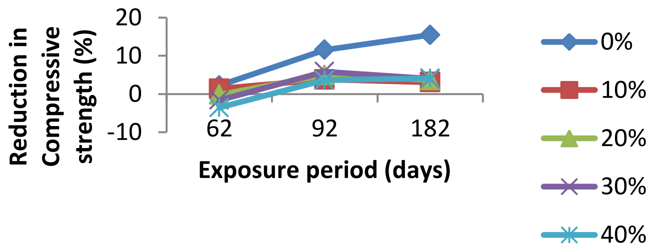

Fig. 9. Reduction in compressive strength of PSA blended cement concrete exposed to 3% $MgSO_4$ solution

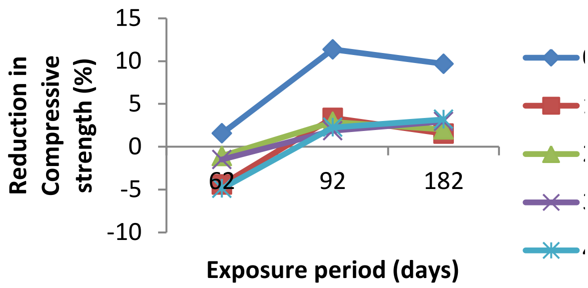

Fig. 10. Reduction in compressive strength of PSA blended cement concrete exposed to 3% Na_2SO_4 solution

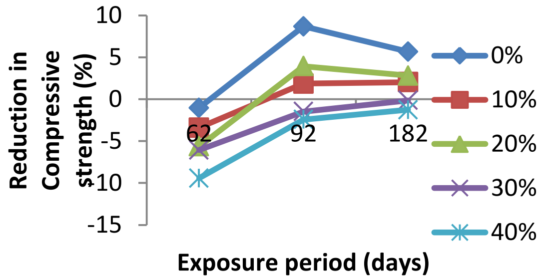

Fig. 11. Reduction in compressive strength of PSA blended cement concrete exposed to 3% $CaSO_4$ solution

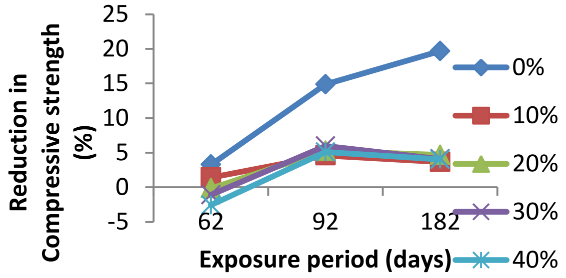

Fig. 12. Reduction in compressive strength of PSA blended cement concrete exposed to 5% $MgSO_4$ solution

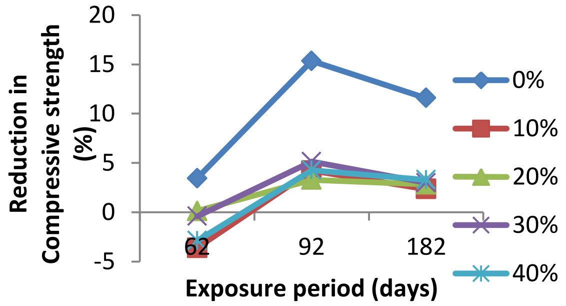

Fig. 13. Reduction in compressive strength of PSA blended cement concrete exposed to 5%

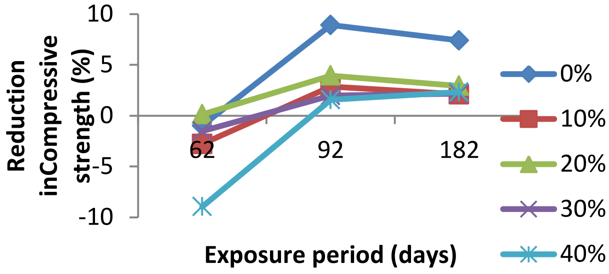

Fig.14. Reduction in compressive strength of PSA blended cement concrete exposed to 5% Na_2SO_4 solution

The statistical analysis with ANOVA as presented in Table 4 indicated that all the factors: percentage PSA content, exposure period, sulphate type as well as sulphate concentration (called independent variables) had significant effects on the measured compressive strength (called dependent variable). It was also established that the interaction of the factors, pair wise and collectively, have significant effect on the compressive strength at 95% confidence level; and that whenever any of these factors is varied, the compressive strength of the concrete changes and the degree of interaction is proportional to the magnitude of change.

TABLE IV
Results of ANOVA of effects of Sulphates on Compressive Strength Test

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected model	8182.534[a]	179	45.712	369.757	.000
Intercept	308426.155	1	308426.155	2494785.348	.000
PSA	7443.233	4	1860.808	15051.633	.000
exposure	102.041	2	51.021	412.693	.000
sulconc	68.718	3	22.906	185.281	.000
sultype	33.682	2	16.841	136.224	.000
PSA * exposure	295.080	8	36.885	298.354	.000
PSA * sulconc	88.746	12	7.396	59.820	.000
PSA * sultype	12.067	8	1.508	12.201	.000
exposure * sulconc	65.516	6	10.919	88.324	.000
exposure * sultype	2.088	4	.522	4.221	.002
sulconc * sultype	16.623	6	2.770	22.409	.000
PSA * exposure * sulconc	19.519	24	.813	6.578	.000
PSA * exposure * sultype	9.469	16	.592	4.787	.000
PSA * sulconc * sultype	12.143	24	.506	4.093	.000
exposure * sulconc * sultype	3.828	12	.319	2.580	.003
PSA * exposure * sulconc * sultype	9.782	48	.204	1.648	.006
Error	44.506	360	.124		
Total	316653.195	540			
Corrected Total	8227.041	539			

The coefficient of determination (adjusted R^2) was 0.992 (99.2%). This implies a strong statistical association between the four independent variables and the dependent variable. Independent variables were estimated to account for 99.2% of the variance in the compressive strength of blended cement concrete cubical specimens. The coefficient of correlation was obtained as R = 0.996. This shows that a very strong linear relationship exist between the two sets of variables being considered.

4. CONCLUSIONS

The following conclusions have been drawn from the study:

1. The compressive strength generally decreases with increase in the PSA content, and PSA content ≤ 10% met the design strength of 25N/mm^2 at 28 days hydration period.
2. The value of the strength loss increases as the concentration of the sulphate increases from 1% to 5% concentration, and exposure period from 62 days to 152 days.
3. Magnesium sulphate solution at 5% concentration and 152 day exposure had greater effect on the compressive strength reduction of both plain and PSA blended cement concrete than other variables.
4. It was statistically observed that the sulphate type, sulphate concentration, PSA contents and exposure period, each and collectively had effect on the concrete compressive strength.
5. Further study should be extended to evaluate the resistance of PSA blended cement concrete in sulphate environment to include the change in mass, change in length and observation of physical appearance of specimens up to 24 months adopting cyclic exposure.

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