

# SULPHATE INDUCED STRENGTH LOSS INDEX AND CALCINATION EFFECT ON PERIWINKLE SHELL ASH POZZOLANA CONCRETE

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**Abstract**— Due to high CO<sub>2</sub> emission, production cost, and energy demand associated with the production of cement amidst its high demand, research on cement alternatives has so far centered on the partial replacement of cement with various materials classified as supplementary cementitious materials (SCM). The favorable prospects of employing SCM's in concrete has since enabled its widespread adoption in many parts of the world notably due to its introduction improving the properties of concrete. However, the use of SCM's does not come without its own concerns. Sustainability demands that the adopted material be available in good amount, and is appreciated if its acquisition and production cost is on the low side. With a view to develop a sustainable binder capable of supplementing cement in concrete, experimental research utilizing agro-based pozzolan; Periwinkle Shell Ash (PSA) in the production of concrete has been conducted with the effect of production variables like calcination temperature and replacement level on concrete's interaction with sulphate-rich environments investigated and reported herein. Observation at 28days curing in a 5% sodium sulphate solution reduced the control strength from its 27.74N/mm<sup>2</sup> to 23.0N/mm<sup>2</sup>, a 17% SISLI. Interestingly, rather than a loss, a gain in strength was observed for all PSA pozzolana concrete at all but 50% cement replacement level. In favour of concrete's resistance to sulphate attack, 400°C was observed to be more ideal calcination temperature amongst the set data gaining a negative SISLI of 37%, 22% and 5% for respective cement replacement levels of 20%, 30%, and 40%. Accordingly, the use of optimally calcined PSA in concrete is an ideal approach in improving concrete's resistance to attack from a sulphated environment.

**Index Terms**— SISLI, Pozzolana Concrete, Calcination Temperature, Strength Activity Index, Sustainability

## 1 INTRODUCTION

A reduction in material lifetime is one of the costliest factors in the construction field [5] hence, the durability of concrete structures is often in doubt when exposed to chemically aggressive environments. One of such chemical attacks is sulphate attack or corrosion which is one of the most frequent and detrimental processes [1] resulting in deterioration of concrete's physical structure. [2] relays that sulphate attack is caused by the formation of ettringite which eventually results in loss of strength, spalling of surface layers and, ultimately,

disintegration. [2] further describes sulphate attack as one that occurs when sulphate-rich soil or water chemically interact with calcium aluminate hydration products in the cement paste formed by the hydration of Portland cement. On investigation of methods with which sulphate's corrosive effect can be reduced, [2] noted that increasing the cement content in concrete would ultimately reduce the rate of concrete's deterioration but not eliminating it. However, increasing the amount of cement used would result in an increase in demand which

relative to its deleterious effect on the environment has raised global concerns. These effects include the emission of carbon

pozzolan with  $\text{Ca}(\text{OH})_2$  and water [8]. [9] further adds that the pozzolanic activity of a material primarily depends on the

S/N	AUTHOR (S)	HEAT TREATMENT		SIEVED THROUGH ( $\mu\text{m}$ )	BASIC OXIDE COMPOSITION (%)					RECOM-MEND. LEVEL (%)
		Calcined ( $^{\circ}\text{C}$ )	Time (mins)		$\text{SiO}_2$	FAS	CaO	MgO	$\text{SO}_3$	
1	Salau et al (2012)	700	90	150	58.0	72.2	8.53	5.02	2.18	15
2	Owolabi et al (2015)	-	-	75	47.2	60.1	8.37	3.8	1.96	5
3	Raheem et al (2015)	X+600	60	150	36.8	46.6	8.2	2.9	1.52	7.5

FAS =  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$

x = Uncontrolled mode of combustion and in the presence of oxygen

dioxide ( $\text{CO}_2$ ) with the production of cement accounting for 6% of the global  $\text{CO}_2$  emission [3]. [3] further adds that the cost increment in energy due to cement production, natural resources depletion, and enough of a supply of raw materials needed in the manufacturing of cement in a desired quality are other major concerns of most cement producing countries. Amidst these concerns, research on cement alternatives has so far centered on the partial replacement of cement with different materials classified as supplementary cementitious materials. Using high-density polythene (HDPE) as partial substitute for fine aggregates, [4] noted that an increase in HDPE content resulted in a reduction in voids and ettringite which led to an increase of sulphate resistivity. This asserts to [2]'s finding that concrete's deterioration rate and durability is inversely proportional to its permeability which is dependent on the w/c-ratio. However, the use of HDPE as SCM cannot be classified as sustainable as it is made from petroleum which is a non-renewable resource. [5] investigated the resistivity of sulphate attack on concrete using SCM's ranging from hydraulic to pozzolanic and then concluded that while slag resulted in higher resistivity, it was only so when applied in large quantities, and analysis showed that cements with a lower content of CaO performed better in resisting sulphate attack. [6] added that calcium content lower than 20% CaO was effective in mitigating expansion while a higher amount of CaO resulted in increased expansion. The findings of [5] and [6] consequently introduces pozzolans, naturally available and consists of very fine particles of siliceous and aluminous materials that in presence of water react with  $\text{Ca}(\text{OH})_2$  to form cementitious materials [8]. The extent of pozzolanic activity is measured with respect to the extent of reactivity of fine dispersion of

amount of  $\text{Ca}(\text{OH})_2$  available for the reaction with the pozzolan, and the reaction rate at which this reaction occurs. The reaction rate depends on physical properties such as the surface area of the pozzolan, the w/c-ratio, and the temperature

TABLE 1  
REVIEWS ON THE USE OF PERIWINKLE SHELL ASH IN CONCRETE

[9]. In observation of research, [9] concluded that increasing calcination temperature resulted in an increase in the particle size of the resulting ash with high pozzolanic activity observed between temperatures ranging from  $500^{\circ}\text{C} - 700^{\circ}\text{C}$ . This temperature range is also acknowledged by [12] with the active stage of the pozzolan observed to occur between  $600^{\circ}\text{C} - 800^{\circ}\text{C}$ . [10] added that a higher fineness of both clinker and calcined clay, an early reactive pozzolan, considerably improves the compressive strength of pozzolana concrete at all ages, particularly at early ages due to the hydration of cement subsequently producing a primary C-S-H gel [11] added to the reaction between  $\text{Ca}(\text{OH})_2$ , a soluble filler, and a pozzolan in other to form a secondary C-S-H gel [2] hence providing pozzolana concrete with even more strength at an early age. In reminiscence of the idea of sustainability when choosing the ideal construction material, and its production conditions, to effectively replace/supplement cement in concrete, researchers have delved into the use of agro-based pozzolans, a much more abundant and readily available material, to partially replace cement with [13]. Agro-based pozzolans are an ideal choice to replace cement with acquitting that they bear resemblance in composition of the four distinct chemical elements, CaO,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$ , of Portland cement clinker built at

a very high calcination temperature. Particularly, periwinkle

## 2 EXPERIMENTAL INVESTIGATION

S/N	TEST TYPE	TEST S.T.D	CUBE COMPUTATION						NO. OF CUBE SPECIMENS
			A	B	C	D	E	F	
1	Particle Size Distribution for Fine Aggregates	[17]							
2	Particle Size Distribution for Coarse Aggregates	[17]							
3	Fineness Test	[18], [19]	1		5			1	5
4	Specific Gravity	[20]	1		5			1	5
5	Water Demand	[21]							
6	Compressive Strength	[22]	1	4	5	1	3	9	69
7	Sulphate Attack	[23], [24]	1	4	5	1	3	9	69
TOTAL									148

N/B: A = Synergy, B = Replacement Level, C = Calcination Temperature, D = Curing Ages, E = Specimen per Age, and F = Control

shell ash (PSA), a product of periwinkle, is observed to be composed of 55.53% of CaO compared to 65.52% composition in Portland cement [14]. In investigation of the use of periwinkle shell ash to supplement cement, [15] concluded that although PSA contained a lower percentage of these chemical constituents, it still is suitable to serve as a replacement for cement. [16] investigated the performance of periwinkle shell pozzolana concrete when subjected to sulphate attack and concluded that the optimum replacement level for which the minimum compressive strength loss was observed was at 10% periwinkle shell ash replacement. Whilst several researchers have investigated the performance of periwinkle shell ash pozzolana concrete and have concluded about varying optimum replacement levels, compressive strength has been observed to increase with curing age and decrease with increasing periwinkle shell ash content.

Having established the independent roles played by calcination temperature and particle fineness on the resistivity of concrete to sulphate attack, this research will be the first of its kind to measure the variations in calcination temperature on fineness and its effect on the sulphate induced strength loss index when periwinkle shell ash is blended with portland cement in concrete.

### 2.1 Materials

2.1.1. Cement. Standard Portland cement, meeting requirement of BS 12:1996, was utilized. Specific gravity and fineness of cement were 3.15 and 2750cm<sup>2</sup>/g, correspondingly.

2.1.2. Fine Aggregate.

2.1.3. Coarse Aggregate.

2.1.4. Periwinkle Shell.

The samples sourced from the surrounding of the main market in Amassoma community, Bayelsa state, Nigeria were washed

TABLE 3  
TESTING METHODS

and sun dried for a 48-hours period to remove organic matter and trapped moisture before being pulverized with a hammer mill in samples passing 600microns.

### 2.1.5. Periwinkle Shell Ash (PSA)

In phase 1, the already pulverized periwinkle shell was calcined in a furnace in the absence of oxygen at an approximate heating rate of 10°C per minute, plus an additional 30 minutes for uniformity. Periwinkle shell was calcined at four temperature levels; 200°C, 400°C, 600°C, and

800°C. In phase 2, the calcined periwinkle shell ash was grinded to sample size passing 90-micron sieve.

TABLE 2  
PRODUCTION TEMPERATURES FOR PSA POZZOLANA

**2.1.6. Sodium Sulphate (Na<sub>2</sub>SO<sub>4</sub>)**

Sulphuric acid was procured from an open market and a concentration of 2.5% of its solution was prepared in the laboratory for the purpose of specimen curing. A control medium (0% sulphate solution) was also created.

**2.1.7. Water**

The experimental work was conducted using water conforming to the specifications of BS 3148:1980.

**2.2. Testing Program**

In other to carry out this experimental investigation, an M20 concrete grade having a nominal mix ratio of 1:2:4, and a constant water/binder-ratio of 0.55 was used. Curing age was observed at 28 days as focus was based off the earliest standardized and approvable age for both local and global adoption. Table 3 summarizes the details of concrete tests carried out for this investigation, and the number of concrete cubes involved.

**3 RESULTS AND DISCUSSIONS**

Fig. 1 represents the particle size distribution of the fine aggregate which shows that the fine aggregate (river sand) used for the experimental study, falls under Zone 3 this class of sand alongside zone 2 are suitable for concrete works [66].

Fig. 2 represents a well graded coarse aggregate suitable for use in concrete with reduced permeability and porosity.

**3.1. CALCINATION EFFECT AT VARIUS TEMPERATURES**

**3.1.1. Color Modification of Periwinkle Shell Ash (PSA)**

Fig.3 shows the effect of calcination temperature on the coloration of PSA samples. From Fig. 3, the coloration of PSA is evidently altered from a light carton color at ATM (25°C) to dark grey at 800°C.

**3.1.2. Fineness Modification of Perwinkle Shell Ash**

Fig. 4, shows the effect of calcination temperature on the fineness of PSA. The control cement sample had a fineness of 62.5% passing the 90micron sieve. However, PSA produced at ambient temperature considerably fell short compared to the

SAMPLE ID	CODE	CALCINATION TEMPERATURE (°C)				
		25°C	200°C	400°C	600°C	800°C
CNTRL	1					
PSA	100P	2	3	5	5	6

control sample with it being 70.4% less than the control sample. The result further reveals that when calcination temperature is increased, fineness also increases noting a direct relationship between both variables. Fineness at 600°C was considerably higher than that of 400°C by 84.4% indicating that the pozzolanic properties of PSA are activated at temperatures around 600°C and beyond. PSA fineness peaked at 800°C with a fineness of 46.49% passing which is only 25.6% less than the control sample.

**3.1.3. Specific Gravity of PSA at Calcination Temperatures**

Fig. 5, shows the effect of calcination temperature on specific gravity of PSA. A parabolic effect was observed with increasing specific gravity peaking between 400°C and 600°C and decreasing at temperatures beyond that. The result revealed that at temperatures between 400°C and 600°C, the average specific gravity (2.96) which was higher than the average specific gravity at ATM (2.66), 200°C (2.78), and 800°C (2.85) was less than that of the control cement sample (3.13) indicating that that a greater amount of cementitious material will result from mass replacement. It is important to note that the production of higher amount of cementitious material would require a higher water-binder ratio to achieve hydraulicity indicating a negative implication on the strength gain capacity of concrete hence whilst a greater amount of fineness was observed at 800°C, specific gravity curve indicates a quadratic relationship with reduction observed at temperatures above 600°C

**3.1.4. Effect of Calcination Temperature on the Water Demand of PSA pozzolana Concrete**

Fig. 6 shows the effect of calcination on the slump value of PSA pozzolana concrete mix when cement is replaced at 40% and 50% respectively. From Fig. 6, slump is observed to increase with increasing calcination temperature however dropping at 800°C, a similar trend to that of the specific gravity. It is important to note that water-binder ratio is directly propor-

tional to slump and inversely proportional to water demand.

### 3.1.5. Compressive Strength of PSA Pozzolana Concrete

Fig. 7 shows the effect of calcination on the compressive strength of PSA pozzolana concrete. From results, compressive strength is observed to be directly proportional to calcination temperature and inversely proportional to replacement level. At 28 days of curing, the compressive strength of the control specimen was 27.74N/mm<sup>2</sup> while compressive strength of PSA pozzolana concrete was observed to peak at 50% replacement level at 800°C with strength ranging from 22.95N/mm<sup>2</sup> at 20% replacement level to 17.86N/mm<sup>2</sup> at 50% replacement level. However, on comparison of the results obtained at 800°C to the control specimen, only specimens produced at replacement levels of 20% and 30% replacement level met the strength activity index with mean 28-day compressive strength of 22.95N/mm<sup>2</sup> and 22.55N/mm<sup>2</sup> respectively. It is also noteworthy that specimens produced at 600°C and 20% replacement level met the strength activity index.

### 3.1.6. Sulphate Attack Resistivity of PSA Pozzolana Concrete

Fig. 8, shows the effect of calcination temperature on the compressive strength of PSA pozzolana concrete after immersion in sulphate solution. From the result, compressive strength of concrete was observed to increase with increasing calcination temperature, peaking at 400°C and then decreases with increases further calcination for 20% and 30% replacement levels, and reduce with increasing calcination temperature, reaching its trough at 400°C and then increases with increasing calcination temperature for 40% and 50% replacement levels. Conclusively, the relationship between sulphate resistivity and calcination temperature is noted to have a parabolic nature. From the results, optimum results were obtained at PSA calcination temperature of 400°C at 20% replacement with compressive strength exceeding that of the control sample by 20.41%. It is also noteworthy that PSA can effectively replace cement up to 50% with specimens produced at 600°C and 50% replacing having a mean compressive strength of 22.07N/mm<sup>2</sup> after immersion which met the strength activity index. Also, PSA pozzolana concrete was observed to gain strength even after immersion as the compressive strength of specimens with optimum results were observed to increase with 37.5%. This alteration maintained the parabolic nature earlier observed for each replacement level.

### 3.1.7. Sulphate Induced Strength Loss Index of PSA pozzolana Concrete

Fig.8 represents the relationship between calcination temperature and the sulphate induced strength loss index of PSA pozzolana concrete. The sulphate induced strength loss index is a percentile measure of the difference in compressive strength before and after immersion representing PSA pozzolana concrete's ability to resist sulphate attack. The results show a decrease of the index with increasing calcination temperature, reaching its trough at 400°C, and then increasing with increasing calcination temperature for cement replacement levels of 20% and 30% respectively while the index is observed to increase with increasing calcination temperature, peaking at 400°C, and then decreases with increasing calcination temperature. Optimum result was obtained at concrete specimens produced at 400°C at 20% cement replacement with compressive strength gain of 37.59%.

## 4. CONCLUSION

Based on experimental tests, the following conclusions were drawn:

1. When subjected sulphate-rich environments, PSA pozzolana cement can effectively resist the corrosive effect of sulphate attack hence it contributes positively to concrete durability.
2. Compressive strength testing has revealed a considerable influence of periwinkle shell ash in concrete production with optimum replacement level at 20% and optimum calcination temperature range; 600°C – 800°C notably lower than that of portland cement production temperature (1300°C – 1450°C).
3. Periwinkle shell ash improves the durability of concrete to a great extent after exposure to sulphate-rich environments notably with its introduction causing concrete to gain strength at 28 days after exposure.
4. When exposed to sulphate-rich environments, periwinkle shell ash can effectively replace 50% of cement content when PSA is produced at 600°C.

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than, - an undergraduate research student with a commendable dedication for hardwork and productivity.

### 6. Conflict of Interest

We the authors of this article titled 'SULPHATE INDUCED STRENGTH LOSS INDEX AND CALCINATION EFFECT ON PERIWINKLE SHELL ASH POZZOLANA CONCRETE' herein state that we are not in any conflict of interest be it financial or otherwise with ourselves or with any external third party. We hereby affirm the absence of any conflicting interest relating with the production and publication of this article.

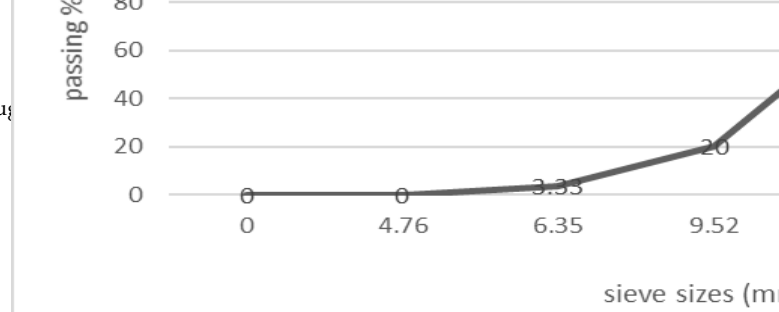


Fig. 2 Particle size distribution of the coarse fine aggregate

## 6 DESCRIPTIVE STATEMENT OF RESULTS

### 6.1 Figures

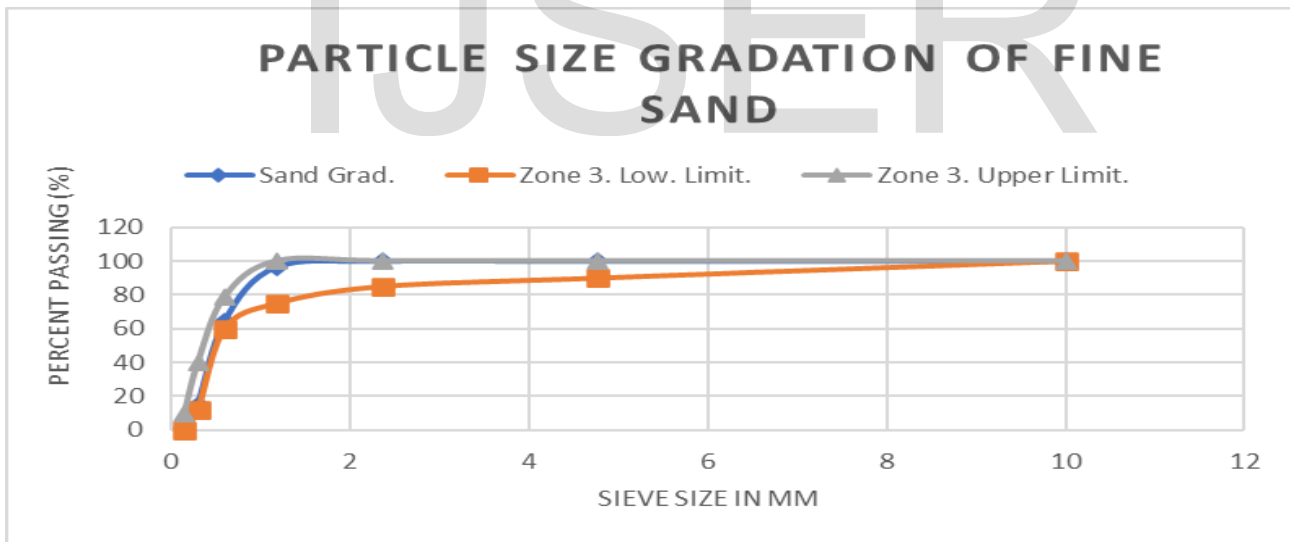


Fig. 1 Particle size distribution of the fine aggregate

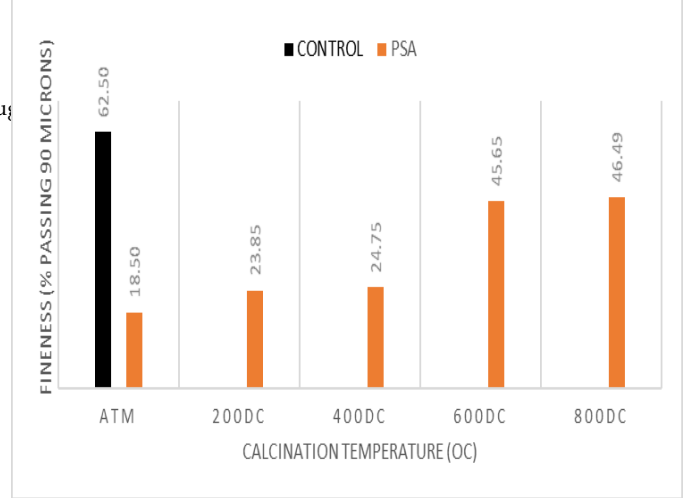


Fig. 4 Effect of Calcination on the Fineness of PSA pozzolana



Fig. 3 Effect of Calcination on the Color of PSA pozzolana

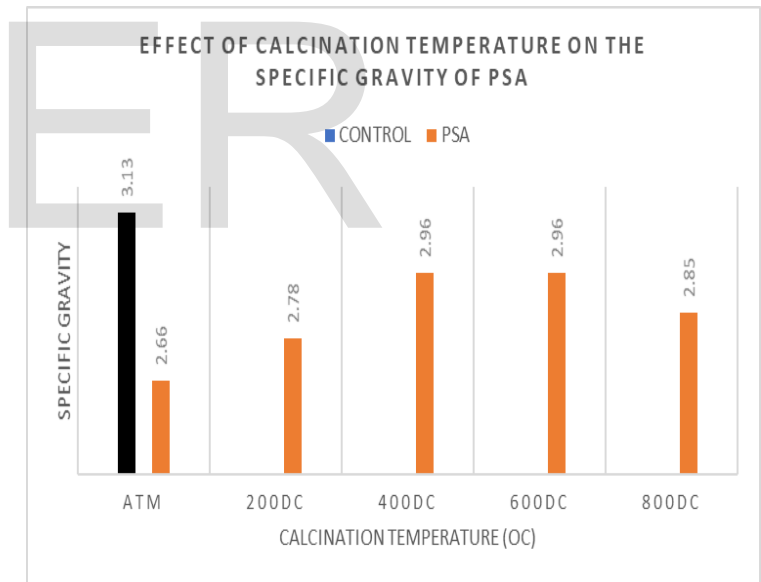


Fig. 5 Effect of Calcination on the Specific Gravity of PSA pozzolana

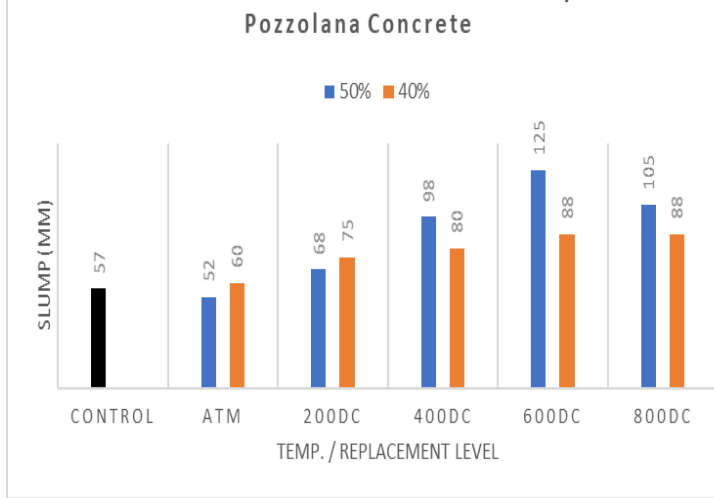


Fig. 6 Effect of Calcination on the Slump of PSA pozzolana

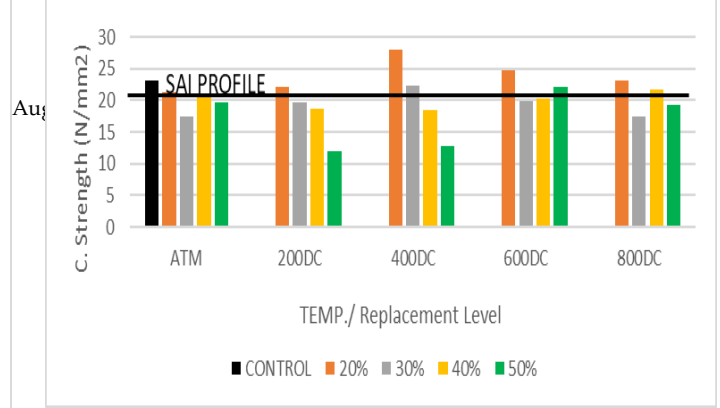


Fig. 8 Effect of Calcination on the Compressive Strength of PSA pozzolana Concrete Cured in a 5% sulphate media for 28 days post hydration period

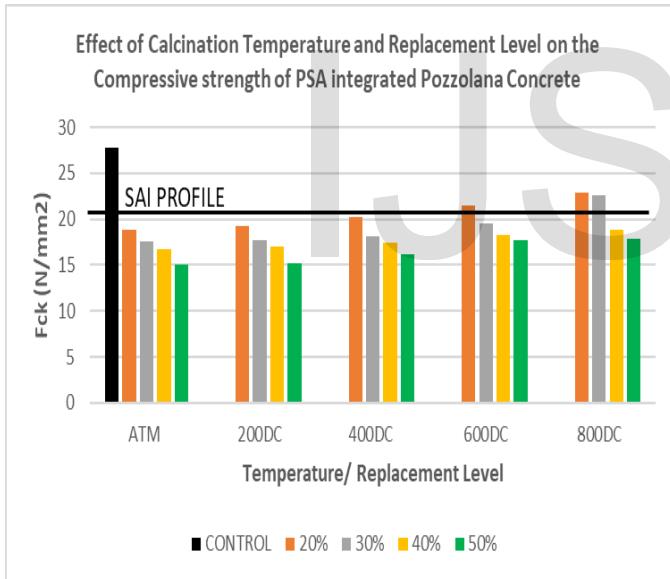


Fig. 7 Effect of Calcination on the Compressive Strength of PSA pozzolana Concrete

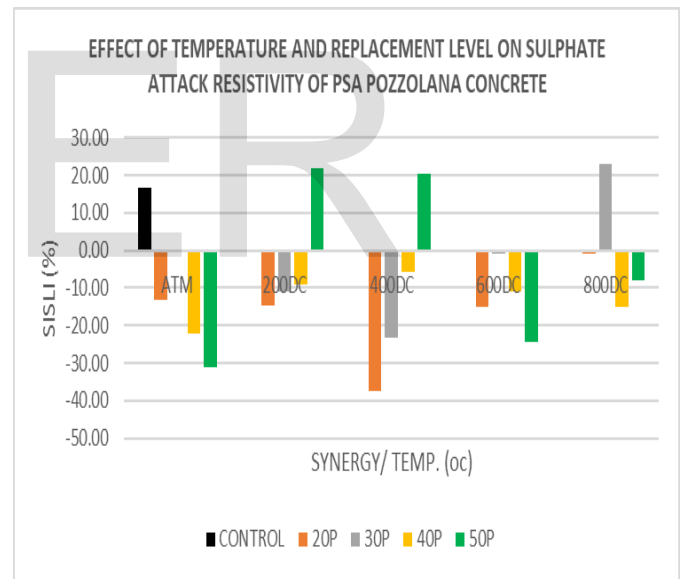


Fig. 8 Effect of Calcination on the Sulphate Induced Strength Loss Index (SISLI) of PSA pozzolana Concrete

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