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Optimization And Impact Of Ternary Blended Cement On Mechanical Strength Durability And Microstructural Studies Of Concrete

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Abstract
This paper investigated the influence of waste materials processed and calcined to activate the cementitious materials for concrete performance. It considered metakaolin (MK) and ground granulated blast furnace slag (GGBFS), as ternary cemetitious materials to partially replace limestone Portland cement (LPcem) in concrete production. The study characterized the cementitious materials, optimally use ternary blended cement to evaluate the mechanical strength of concrete at 7, 14, 28, 60 and 120 days, applied heat at elevated temperature of 150^{0}C for 6 hours duration to determine the percentage weight, length change and microstructural analysis of the concrete samples for 28 and 60 days. Experimental results demonstrated that ternary blended cement improved strength, at 28 days compressive strength the percentage increase of 23.8% and 17% to mix 1 and mix 2 (controls). Flexural and split tensile strength increases strength throughout the ages. Evidence of weight and length changes loss during the 6 hours duration of elevated temperature. Microstructural analyses as shown on the energy dispersive spectrometer (EDS) spectra confirmed element composition of O, Ca, Al, Si, Na etc. Voids, cracks and improved pore structure with hydration properties with calcium hydrate gel (C-H) in limestone Portland concrete (LPC) and calcium silicate hydrate gel (C-S-H) in partial replaced concrete (PRC). These findings highlight the potential of optimal utilization of blended cement as sustainable supplementary cementitious materials for concrete development.
Keywords: *Ternary blended cement, mechanical strength, characterization, elevated temperature, and microstructure*

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I. Introduction

The demand for cement in concrete and other infrastructural facilities is ever increasing day by day. Concrete consist of cement, fine- coarse aggregates and water to form concrete, it may be reinforced and plain, which demands a considerable properties such as compressive, flexural strength and split tensile strength to give satisfactory durability during its service life (PCA 2011). Shobeiri *et al.,* (2021) reported that the main constituent, cement is responsible for the emission of 7% carbon dioxide (CO_2), the total global CO_2 gas, the key green-house gas held responsible for the global warming and climate change. Each ton of portland cement produced releases 1 ton of CO_2 (Bakhtyar *et al.,* (2017). To mitigate this, an industrial, natural occurring materials or the combination of this waste by-product (ternary blended) when processed contain supplementary cementitious materials or pozzolans which can partially replace cement in concrete infrastructure.

Khalil and Anwar, (2015) tested blended cement concretes and reported that there are technical, economic, and environmental benefits using ternary blended cements. Aktham *et al*., (2019) opined that ternary blends can even be more durable than binary blends because the additional SCMs can overcome the shortcomings of the second SCMs. However, it should be noted that the properties do not follow a superposition behavior. The SCMs may interact with each other, and properties found in ternary blends may differ from those found in binary blends. Yi Han, *et al.,* (2021) performed binary and ternary blended cements for resistance to alkali silica reaction (ASR), the study concluded that binary combinations of silica fume and Portland cement had increased alkalinity at ages beyond 28 days. However, ternary blends of Portland cement, fly ash, and silica fume maintained low alkalinity for up to 3 years and kept expansion below 0.04%. Thomas (2016) studied a long-term test on the durability of ternary blended cements using GGBFS and silica fume, the study consisted of laboratory and outdoor exposure site testing. Slabs of different combinations were cast in harsh environments. Yi Han, *et al.,* (2021) tested binary and ternary combinations of Portland cement, GGBFS, and limestone for strength gain. They concluded that binary combinations of portland cement and GGBFS had low early age strength, but higher strengths at later ages. Binary combinations of portland cement and limestone had high early strengths, but reduced later age strengths. However, ternary combinations of portland cement, limestone, and GGBFS had both high early age and later age strengths. Thiago *et al*., (2021) did a similar study on strength gain using Portland cement, GGBFS, and metakaolin. They also found that ternary blends of the materials maintained the benefits of each binary combination while overcoming any short comings the binary blend experienced.

This work is an investigation to use ternary blended materials, such as limestone Portland cement (LPcem), metakaolin (MK), and ground granulated blast furnace slag (GGBFS) which are natural source materials and industrial by–product. Characterize the cementitious materials, optimize and evaluate the performance of ternary blended concrete using mechanical strength test, elevated heat and microstructural study.

II. Materials

Plate 1: (a) metakaolin, (b) GGBFS and (c) LPcem

III. Methods

X-ray difractometer (XRD) and X-ray fluorecence

Metakolin (MK), ground granulated blast furnace (GGBFS) are calcined and characterised by X-ray difractometer (XRD) and X-ray fluorescence (XRF) respectively. The XRD determines the mineralogy; XRF test shows the chemical composition and the (SEM) scanning electron microscope test determines the microstructural analysis of the cementitious materials.

Table 1: Optimized mix percentage of limestone Portland cement LPcem and the blended cement

Mix No.	% LPcem	% - MK	% - GGBFS
1.	100	0	0
2.	100+conpl	0	0
3.	90	5	5
4.	80	15	5
5.	80	10	10
6.	80	5	15
7.	70	20	10
8.	70	15	15
9.	70	10	20
10.	60	30	10
11.	60	20	20
12.	60	10	30
13.	50	30	20
14.	50	25	25

The materials are limestone Portland cement (LPcem), supplementary cementitious materials (SCM) as metakaolin (MK) and Ground granulated blast furnace slag (GGBFS). Table 1 showed the full exploitation and optimization of the cementitious materials mix 1 to 14. Tables 2 consist of the exploited SCMs, partially replace cement mixed with fine, coarse aggregates, water with addition of sulphonated naphthalene admixture of 1.5% (conplast SP430) to increase fluidity and workability of the concrete. Mix 1 and 2 is limestone Portland concrete (LPC) as control while mix 3 – 14 is partially replaced concrete (PRC).

Plate 2: (a) Cast (b) cured samples respectively

Mix Proportion of Ternary Concrete

Table 2: Optimised mix proportion of ternary blended concrete and 1.5% conplast

Mix No.	LPcem kg/m^3	MK kg/m^3	GGBFS kg/m^3	FA kg/m^3	CA kg/m^3	Water kg/m^3	1.5% Conpl. kg/m^3
1	400	-	-	600	1200	190	0
2	400	-	-	600	1200	190	6
3	360	20	20	600	1200	190	6
4	320	60	20	600	1200	190	6
5	320	40	40	600	1200	190	6
6	320	20	60	600	1200	190	6
7	280	80	40	600	1200	190	6
8	280	60	60	600	1200	190	6
9	280	40	80	600	1200	190	6
10	240	120	40	600	1200	190	6
11	240	80	80	600	1200	190	6
12	240	40	120	600	1200	190	6
13	200	120	80	600	1200	190	6
14	200	100	100	600	1200	190	6

Mechanical strength test

Compressive strength test

150 mm cube samples produced from various mixes is tested and determined using the compression machine at 7, 14, 28, 60 and 120 days, according to ASTM C109/C 109 M-12

Flexural Strength test

Flexural strength test of 100 mm by 100 mm by 350 mm beam samples determined using the universal testing machine at 7, 14, 28, 60 and 120 days, according to ASTM C 78.

Split tensile test

Cylindrical mould of size of 150 mm diameter and height of 300 mm of composites produced and tested to determine the split tensile strength using the compression machine at 7, 14, 28, 60 and 120 days, according to ASTM C496.

The 210 numbers each of cubes, beams and cylindrical specimen sizes are cast respectively, made up of limestone Portland concrete (LPC) as control and partially replaced concrete (PRC) adding conplast SP430 an admixture to increase flow ability, workability to enhance the cementitiuos materials in the mix. An effective way to create the less permeable concrete is partly substitution of cement with supplementary cementitious materials (SCM) to increase compressive strength in concrete Pratiwi, *et al.,* (2021). These samples are cured in water, and separately tested for strengths at 7, 14, 28, 60 and 120 days. On each crushing date, three cubes are removed from water weighed and dried. Taken to the universal tensile machine (UTM) crushed and recorded the compressive strength and the other crushing days.

Plate 3: (a) Cube (b) Cylinder and (c) Flexural beam under UTM test

Heat elevation test

Testing of concrete samples at 150^0C (elevated temperature)

The concrete was subjected to high temperature for 6 hours of drying, shrinkage occurs when the specimen are subjected to drying conditions. Limestone Portland concrete (LPC) and partial replaced concrete (PRC) are subjected to a temperature of 150^0C in an oven for 6 hours after 28 days cured in water. Each cube was withdrawn every hour. It is the loss of water held in gel pores that causes the volume changes. The percentage length change and weight loss of the cubes are measured and plotted against hours.

Weight and length of each specimen were measured by weighing machine and Vernier tool Plate 4 immediately taken out of curing tank as total weight (W_T) and length (L_T) respectively. Put inside oven for 6 hours and taken out every hour for measurement of new weight (W_N) and length (L_N).

Percentage weight and Length change of concrete

Length and weight changes test is conducted on the samples in accordance to ASTM C140 and ASTM C-20 at every hour in an oven. Percentage change is measured as Total weight – dry weight divided by total weight multiplied by 100%.

Percentage change in Weight = $\frac{W_T - W_D}{W_T} x \frac{100}{1}$ (1)

Where W_T is the total weight of cube and W_D is the dry weight

Percentage Length change = $\frac{L_T - L_N}{L_T} x \frac{100}{1}$ (2)

Where L_T is the total length and L_N is the new length

Plate 4: Concrete samples subjected to 150^0C elevated temperature for 6 hours

Scanning Electron Microscope and Energy dispersive x-ray spectroscope (SEM/EDX)

The influences of limestone Portland cement, metakaolin, ground and granulated blast furnace slag as a ternary blended cement in concrete sample, was assessed using the scanning electron microscope incorporated with energy dispersive spectroscopy machine (SEM/EDX) in line with Aktham *et al*., (2019). The (SEM/EDX) test is performed at the age of 28 and 60 days, following the guidelines prescribed in (ASTM C1723, 2010).

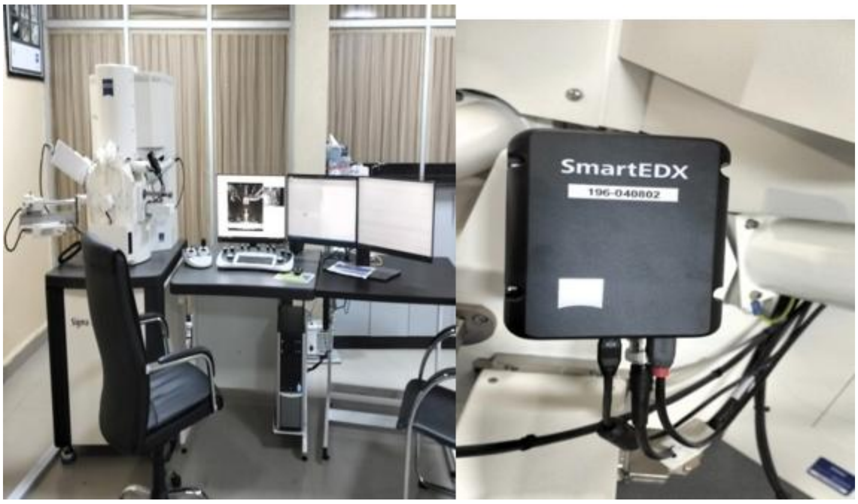
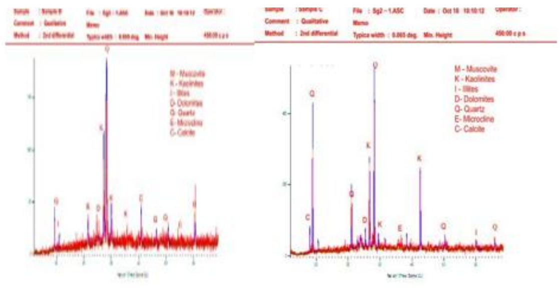


Plate 5: Zeiss Sigma300VP Scanning Electron Microscope with embedded Smart EDX

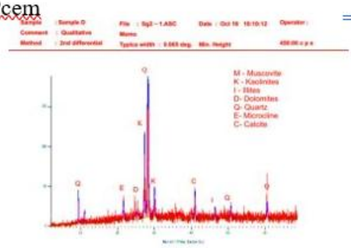
IV. Results And Discussion

Characterization of cementitious materials

Spectra of X –ray diffractometer of cementitious materials



(a) LPcem (b) GGBFS



(c) MK

Fig. 1: XRD spectra of (a) LPcem, (b) GGBFS (c) MK

XRD patterns revealed the crystalline nature of these raw materials as shown in the Fig.1 above respectively. All the raw materials showed the presence of expected mineralogical phase or compound such as quartz, kaolinite, illite, dolomite, muscovite, microcline, calcite etc. Quartz is the major compound with the highest characteristic peaks and peaks corresponding to other compounds observed in the pattern are less in concentration or intensity.

It is seen that the LPcem, GGBFS and MK are partly crystalline due to sharp peaks which indicated their contribution in ternary cement. XRD graphs indicated the crystalline peaks of MK and GGBFS were lower than that of LPCem. However, a prominent diffraction peak observed at 20^0 - 28^0 at 2θ signifies the notable reactivity potential of the pozzolans according to Zhiyuan *et al.,* (2024).

According to ASTM C618 (2001), the code categorized pozzolan less than 70% (<70%) as class C, class F (≥ 70%) and class N (> 70%). The sum total of silicon oxide (SiO_2) + Aluminium oxide (Al_2O_3) + Ferric Oxide (Fe_3O_3) in each are LPCem79.4%, GGBFS 90.5% and MK 90.6% respectively classified them as class N (> 70%) are all pozzolanic materials with the loss on ignition LOI less than 6%. The results obtained were perfectly in line with ASTM C618 as presented in Table 3 and their Al/Si ratio of LPcem 0.2, GGBFS 0.55 and MK 0.52 respectively.

Table 3: Chemical composition of LPcem, GGBFS and MK obtained by XRF analysis

Oxide %		LPcem[A]	GGBFS[B]	MK[C]
SiO_2		62.4	55.4	56.3
Al_2O_3		13.3	30.6	29.2
Fe_2O_3		3.7	4.5	4.95
TiO_2		1.02	1.33	1.25
CaO		0.05	1.65	1.55
P_2O_5		0.08	-	0.04
K_2O		0.96	0.89	0.98
MnO		0.03	0.08	0.08
MgO		0.15	4.69	4.75
Na_2O		0.09	0.87	0.90
LOI		18.4	0.04	0.02
$Al_2O_3+SiO_2+Fe_2O_3$		79.4	90.5	90.6
Al_2O_3/ SiO_2 ratio		0.2	0.55	0.52

Mechanical strength test

Compressive strength of concrete cubes LPC/PRC

Compressive strength of limestone Portland concrete (LPC) and partially replaced concrete (PRC). The 28th days characteristic strength of each mixed are considered, the highest value of compressive strength is noted and recorded. This happened to be mix 7 which recorded 42 MPa 23.8% and 17% increase to mix 1 and mix 2 which are 32 MPa and 35 MPa. Mix 2 was achieved by adding sulphonated naphthalene chemical admixture (conplast SP430), these increase workability and flow ability of the concrete Fig. 2. Fig. 3 and 4 is the modulus of rupture and split tensile strength, 17 and 18% over the control 1 respectively.

Table 4: Average compressive strength of concrete cubes

		Av. Compressive strength (MPa)			
Days	7	14	28	60	120
Mix No.					
1.	25	28	32	35	40.3
2.	26.2	30.3	35	40.6	45
3.	27.8	33.5	37.4	41.4	47
4.	28.6	33.7	38	42.2	48
5.	27.3	30.5	38.4	41.5	49.7
6.	18	22.6	26.7	30.5	35
7.	30.7	34.5	42	45	50.5
8.	26.8	30.9	35.3	40.6	46
9.	19.5	23.6	28.8	31.5	34.5
10.	27.4	32.2	34.9	40.3	43
11.	25.5	30.9	33.4	40.1	42
12.	20	23.9	30.7	35.4	41
13.	19.8	22.1	27.3	30.8	36
14.	18.5	20.3	25.5	28.9	33

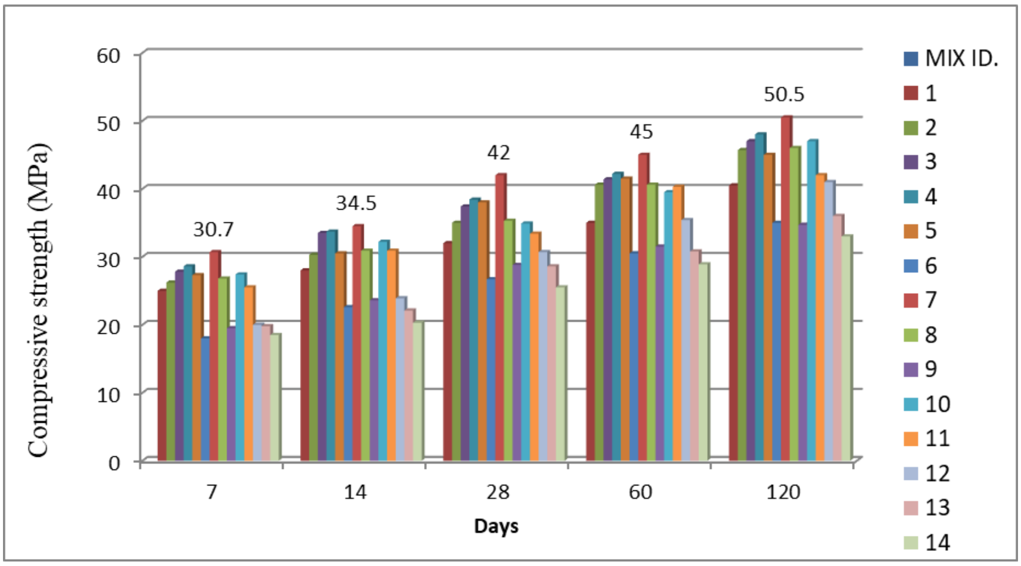


Fig 2: Average compressive strength of cubes

Flexural strength of beams

Table 5: Average Flexural strength of beams

Mix. No.	Average Flexural strength (MPa)				
	Days				
	7	14	28	60	120
1.	4.2	4.7	5.2	5.6	6.5
2.	4.4	4.6	5.6	5.8	6.8
3.	4.8	5.1	5.4	5.9	6.9
4.	4.5	5.2	5.7	6.2	7.5
5.	4.1	4.4	4.8	5.3	6.3
6.	4.0	4.3	4.7	5.5	6.4
7.	5.4	5.8	6.2	6.5	7.8
8.	4.3	4.7	5.3	5.7	6.7
9.	3.5	3.8	4.2	4.7	5.6
10.	5.2	5.6	5.9	6.1	7.2
11.	3.2	3.6	3.8	4.1	5.4
12.	3.3	3.4	3.7	3.9	4.5
13.	2.9	3.2	3.5	3.7	4.3
14.	2.2	2.9	3.2	3	3.9

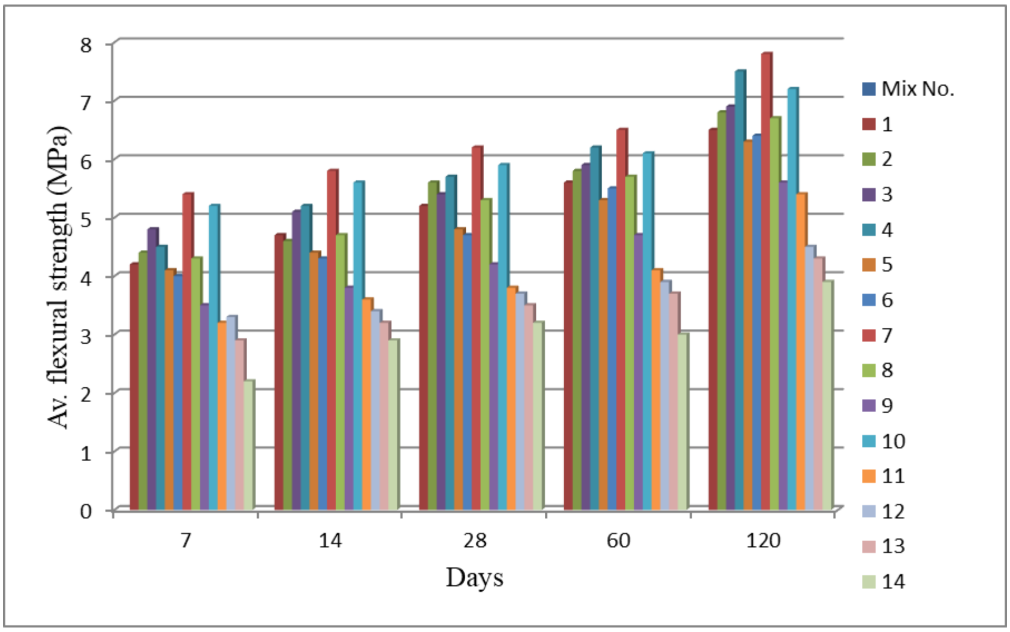


Fig. 3: Average modulus of rupture

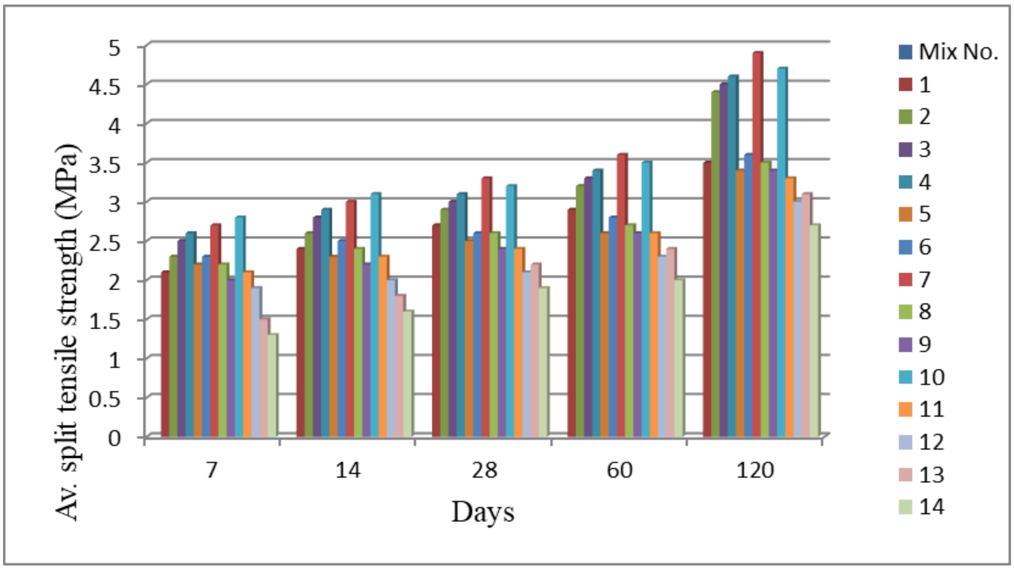


Fig. 4: Average split tensile strength of cylinder

Split Tensile Strength

Table 6: Average split tensile strength of cylindrical concrete

Mix No.	Average Split tensile strength (MPa) Days				
	7	14	28	60	120
1.	2.1	2.4	2.7	2.9	3.5
2.	2.3	2.6	2.9	3.2	4.4
3.	2.5	2.8	3	3.3	4.5
4.	2.6	2.9	3.1	3.4	4.6
5.	2.2	2.3	2.5	2.6	3.4
6.	2.3	2.5	2.6	2.8	3.6
7.	2.7	3	3.3	3.6	4.9
8.	2.2	2.4	2.6	2.7	3.5
9.	2.0	2.2	2.4	2.6	3.4
10.	2.8	3.1	3.2	3.5	4.7
11.	2.1	2.3	2.4	2.6	3.3
12.	1.9	2.0	2.1	2.3	3.0
13.	1.5	1.8	2.2	2.4	3.1
14	1.3	1.6	1.9	2.0	2.7

Impact of concrete subjected to heat elevated temperature of 150^{0}C

Concrete cubes of size 75 mm x 75 mm x 75 mm of LPC, PRC, and GPC was subjected to 150^{0}C in the laboratory oven for duration of 6 hours to know the effects of shrinkage as per the weight loss in percentage and length change Fig. 5 and 6. The effect showed significant weight loss on the concrete samples, the primary effect from 25^{0}C to 100^{0}C had to do with the residual pore water which evaporated from capillary pores and the second 100^{0}C - 150^{0}C on dehydration of calcium silicate hydrate (C-S-H), ettringite and calcium aluminate hydrate (C-A-H) (Reddy and Naqash, 2019). The effect on the LPC was seen higher and decreasing every time the temperature rises, the partially replaced (PRC) also increases but at lower and decreasing rate. The effect on LPC was due to the dehydration of water in the cement gel and decomposition of $Ca(OH)_2$ which weaken the concrete matrix, cracking and spall due to the heat which causes internal pressure that eventually caused durability loss in the LPC. The PRC concrete consist of cementitious materials which protect and reduces the effect of the heat on the cement gel.

Percentage change in weight

Table 7: Percentage change in weight of concrete subjected to elevated temp. of 150^{0}C

	LPC			PRC		
Hrs	Wet wt.	Dry wt.	% wt. change	Wet wt.	Dry wt.	% wt. change
0	1152	1152	0	1151	1151	0
1		1085	94		1115	96
2		1018	88		1039	90
3		956	82		1025	89
4		910	78		1009	87
5		804	70		956	83
6		610	53		674	59

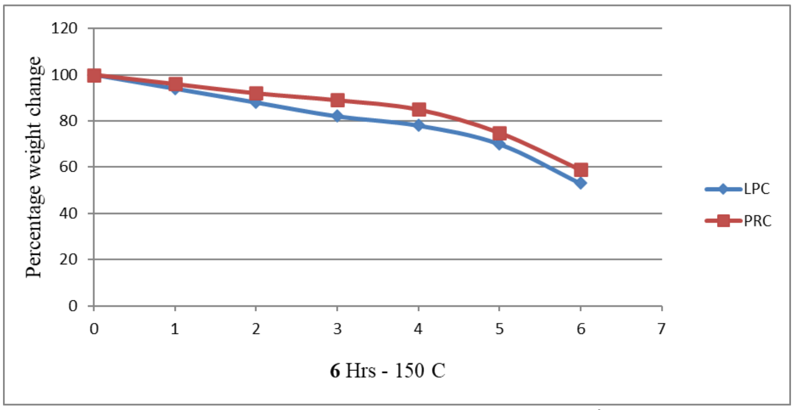


Fig. 5: Percentage weight change when subjected to 150^{0}C (elevated temp.)

Percentage Length change

Table 8: Percentage length change when subjected to 150^0C elevated temp.

Hrs	Original Length (mm)	New length (mm)	% Length loss	Original Length (mm)	New length (mm)	% Length Loss
	PRC			LPC		
0	75	75	100	75	75	100
1		74.8	99.7		73.5	98
2		74.2	98.9		73	97
3		74	98.6		72.5	96.6
4		73.5	98		72.1	96
5		73	97.3		70.5	93
6		72.8	96.5		65	87

Fig. 6 showed how the cubes length % yielded to the temperature increment, the size of the cubes reduces, LPC length crashed from 98% in an hour to 87% in 6 hours, PRC resisted the temperature in an hour of 99.7% to 96.5% in 6 hours This is due to the additives that protected the concrete from the heating the aggregate directly not being touched. The colour becomes whitish, spall, brittles, and cracks became more pronounced in the LPC followed by the PRC as confirmed by (Abed *et al.,* 2021 and Amran *et al.,* 2022).

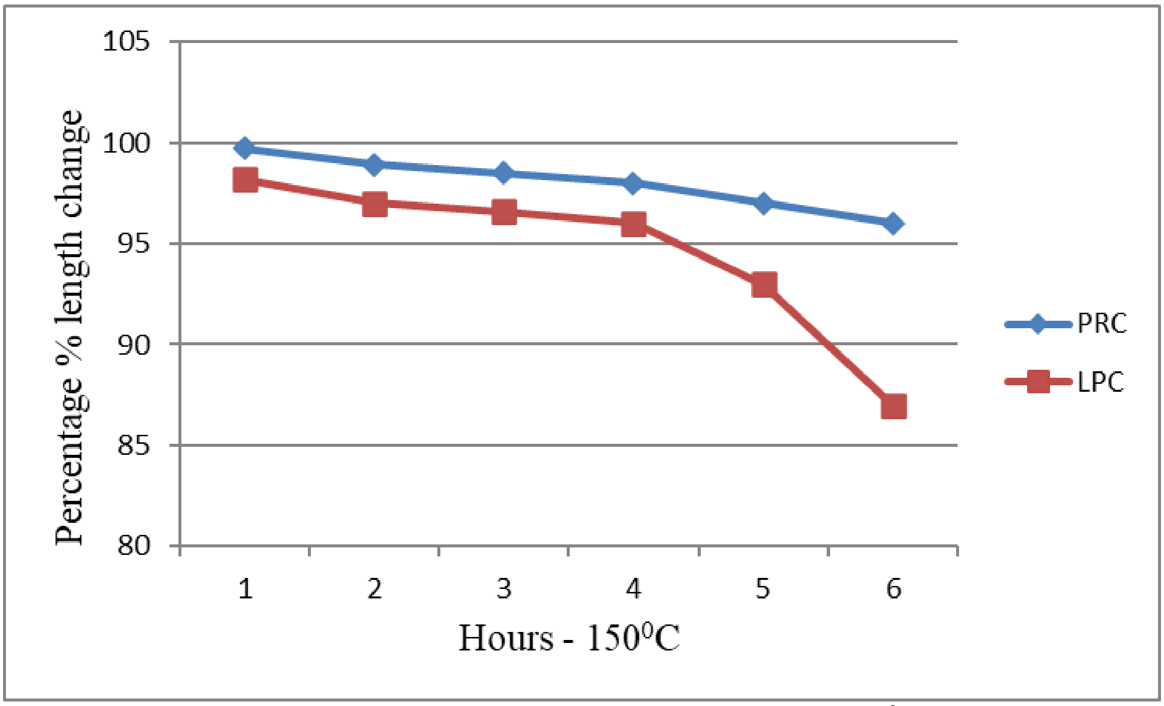
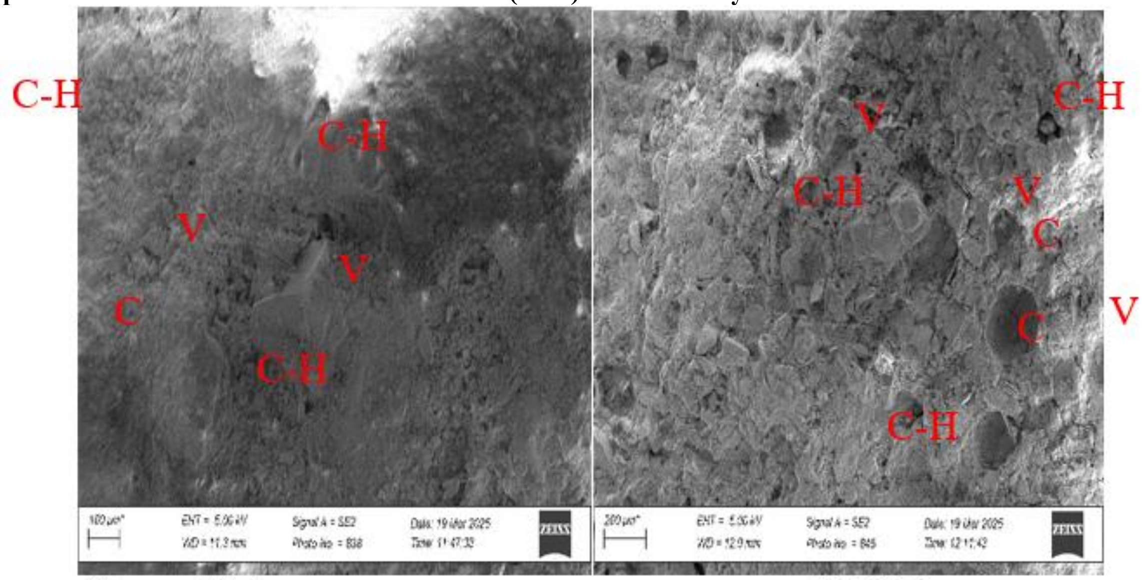


Fig. 6: Percentage change in length at elevated temp. of 150^0C

Morphology test of 28 days and 60 days of concrete sample specimens

SEM analysis gives information regarding the surface morphology or microstructure of the hardened concrete samples. Microstructural analyses confirmed improve pore structure and hydration properties. The water in limestone portland cement (LPcem) combined with the $(CaOH)_2$ in the concrete to formed calcium hydrate C-H Plate 6. The partially replaced concrete (PRC) contained water in LPcem mix with $(CaOH)_2$ and silicate to formed the C-S-H concrete Plate 7. Voids and cracks were seen through the micrographs. The ettringite minerals could permeate the concrete through the voids and cracks created in concrete to allow chloride ion, sulphate ions, CO_2 and acid to attack the concrete which can lead to expansion, contraction and peeling of reinforcement which may compromise the issue of durability in the concrete in agreement with (Sushant, 2020). It is through the voids created that chloride react with the deleterious materials in aggregates to caused alkar silica reaction, Sulphate ions attacked concrete and reinforcement, CO_2 reacted with $CaOH_2$ to cause carbonation degrading the concrete. The elemental EDS spot analysis conducted on the SEM micrograph revealed the elements O, Si, Al, Ca, including other elements suggesting hydrated C-H in LPC and C-S-H in PRC Fig 7 and 8.

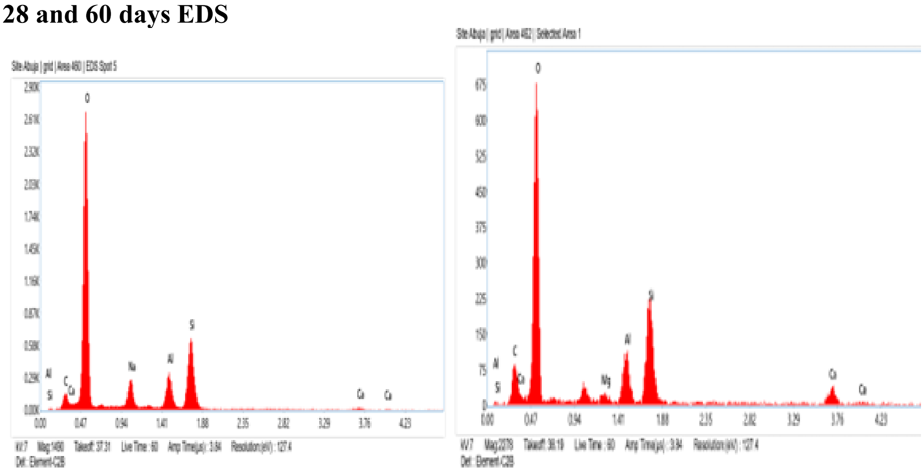
Type of concrete - limestone Portland concrete (LPC) 28 and 60 days SEM /EDS



(a) 28 days (b) 60 days

Plate 6: Micrograph of (a) 28 and (b) 60 days 100% limestone Portland cement concrete sample

100% LPC 28 and 60 days EDS



(a) (b)

Fig. 7: EDS of LPC (a) 28 and (b) 60 days spectra depicting elements peak in the concrete sample

Type of concrete – Partial replaced concrete (PRC) 28 and 60 days SEM

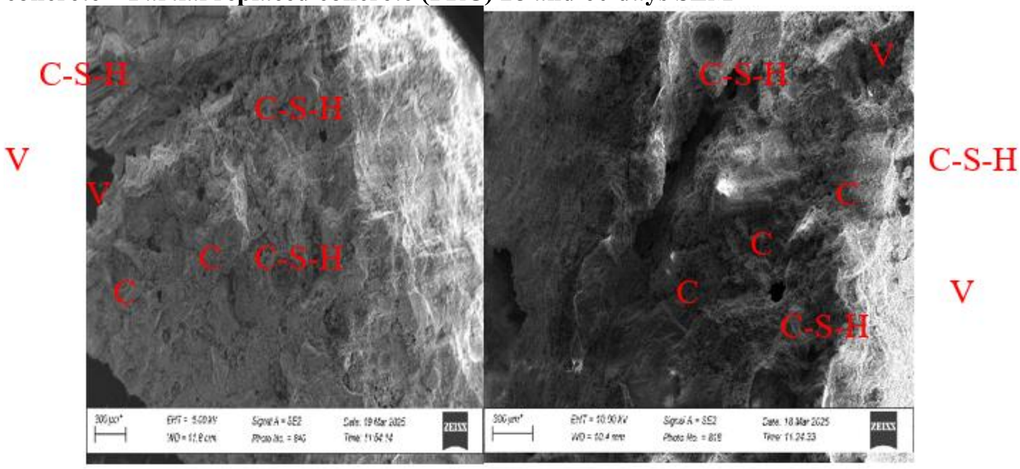


Plate 7: Micrograph of (a) 28 and (b) 60 days 70% LPcem + 20% MK + 10% GGBFS partial replaced concrete

PRC 28 and 60 days EDS

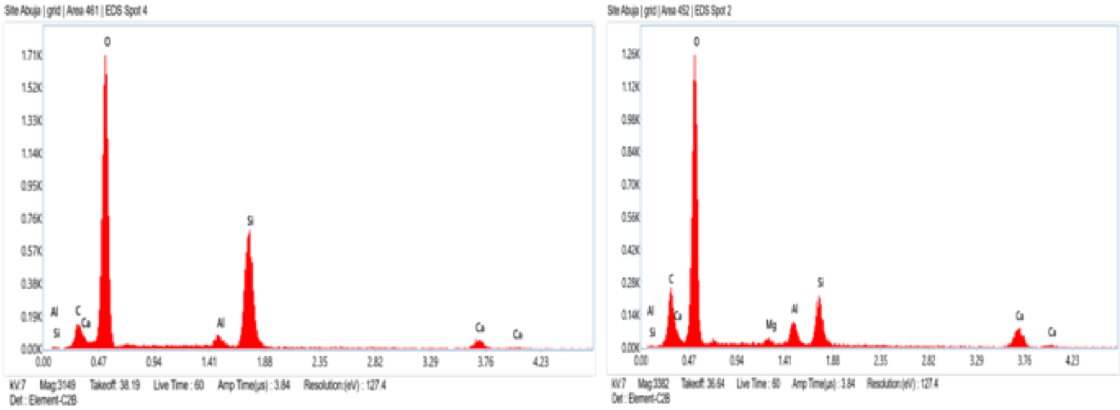


Fig. 8: EDS of PRC (a) 28 and (b) 60 days spectra depicting elements peak in the concrete sample

V. Conclusion

- ❖The XRD patterns of the SCMs revealed the crystalline Quartz, the major compound, due to its thin shape and the height, other compound are less reactive.
- ❖XRF Sum total of $SiO_2 + Al_2O_3 + Fe_3O_3$ in the cementitious materials showed that they are class N i.e > 70% supplementary cementitious materials or pozzolans, made it very reactive and Al/Si ratio of 0.52 workable.
- ❖Optimal use of the SCMs led to the mix proportion of mix 1 -2 as control, 3 – 14 mixtures made the concrete mix to be fully assessed and tested at 7, 14, 28, 60 and 120 days to give different strength of concrete.
- ❖The mechanical strength at 28^{th} days compressive strength test of PRC was 42 MPa, the control of LPC 32 MPa and 35 MPa (mix 1 and mix 2) which was 23.8% and 17% increase over the controls. The flexural strength recorded 17% increase, likewise the split tensile strength of 18% increase over the control respectively.
- ❖Effect of elevated temperature measured on concrete weight for 6 hours, LPC 610 kg at 53% and PRC 674 kg at 59%. LPC length shortened to 65 mm at 87% while PRC 72.8 mm at 96.5% for the 6 hours temperature. Weight and length reduces as the temperature increases, more in LPC than PRC. It showed PRC had good resistance to elevated heat.
- ❖The morphology of concrete samples obtained by EDS test showed some % elements- O, C, Al, Si, Ca etc. Evidences of calcium hydrates C-H compound in LPC and calcium silicate hydrates C-S-H compound in PRC. Voids and cracks are seen in the concrete micrographs, which made the concrete susceptible to some enttrigite minerals such as sulphate ions, CO_2, chloride and acid penetration that may attack the concrete aggregates and reinforcements to cause the degradation of the concrete structures.

VI. Recommendation

The Partially replaced concrete (PRC) samples optimally used cementitious materials performed better in compressive, flexural and split tensile strength, had high resistance to elevated temperature and are much more formidable hardened concrete than LPC.

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