[1.] B. F. AKINBILE, [2.] W. A. JAYEOLA

POZZOLAN REACTIVITY, MINERALOGY AND MORPHOLOGY OF BAMBOO LEAF ASH (BLA) AND PALM FRUIT FIBRE ASH (PFFA) BLENDED CONCRETE

[1.]Department of Building Technology, The Polytechnic, Ibadan, NIGERIA
[2.]Department of Civil Engineering, The Polytechnic, Ibadan, NIGERIA

Abstract: This research investigates the reactivity, mineralogy and morphology of BLA and PFFA and its influence in their replacement with Portland Cement (PC) in blended concrete. Physical properties, chemical compositions, microstructural and mechanical behaviors of BLA, PFFA and the blended concrete were examined. Modified Chapelle test was adopted to determine the pozzolanic reactivity levels of BLA and PFFA while chemical compositions, mineral contents and microstructure of BLA and PFFA were obtained with X–Ray Fluorescence (XRF), X–Ray Diffraction (XRD) and Scanning Electron Microscope (SEM)/Energy–Dispersive X–ray (EDX) analysis respectively. Cubes (150mm x150mm x 150mm) of concrete blends with BLA, PFFA and PC were produced in 0%, 5%, 10%, 15%, 20% and 25% percentage replacement. The compressive strength was determined at 7, 28, 56 and 90 days of hydration. The results revealed that BLA and PFFA has 2353.53mg and 1233.2mg $Ca(OH)_2$ consumption respectively and meet pozzolan materials standards. EDX spectra showed silica in high content for both ashes. Optimum strength of 46.58 MPa was recorded in 10% BLAPFFA blended concrete at 90days. The study therefore concluded that BLA and PFFA were pozzolanic, hence, suitable for partial replacement of PC in concrete production.
Keywords: bamboo leaf ash, concrete, palm fruit fibre ash, pozzolan, microstructure

1. INTRODUCTION

Infrastructural development is on the high rise globally, consequently resulting in an increase in the request for construction materials like concrete, which has been identified as one of the best–used construction materials in supplying the demands for the infrastructure development. McLellan, *et al*. (2011) described Portland Cement (PC) as the major binder in the concrete materials, however, it is very costly; mostly in developing countries in which Nigeria is not excluded. Moreover, Tulashie et al., 2021 noted that cement production is associated with the high energy demand and clinker's calcination high temperatures of about 1450 **°C** with the resultant effect of greenhouse gases like carbon dioxide (CO_2) emission of 0.85 ton per 1 ton of cement manufacturing. After the power generation industry, cement manufacturing industry was rated second–largest contributor of CO_2 emissions (Wi *et al*., 2018), as clinker the cement manufacturing product's annual global CO_2 emissions was estimated to 5–8% (Afroughsabet *et al*., 2021). This necessitates the needs for sourcing other alternative materials capable of reversing this and other environmental phenomenon like global warming and ozone layer depletion by researchers and practitioners. Pozzolan materials originating from industrial and agricultural waste such as, fly ash (FA), Ground Granulated Blast Furnace Slag (GGBS), Silica Fume (SF), Metakaolin (MK), Bamboo Leave (BL), Palm Kernel Shell (PKS), Rice Husk (RH), Saw Dust (SD), Groundnut Husk (GH), Periwinkle Shell (PS), Coconut Shell (CS), etc., has been identified as among the alternative pozzolan materials. The waste generated through agricultural activities can be sourced in tonnes as they are deposited in the environment without effective technique of waste management and reprocessing (Ikponmwosa, et al., 2019). Decomposition and burning of the heaped agriculture waste creates environmental pollution, hence the needs for their recycling and reusable process. The rural dweller where these waste are majorly produce and readily available can maximize its recycling to boost small scale industry in the area hereby turning waste to wealth and reduce poverty if their recycling process and usability is established and standardize.

Pozzolan is a natural or artificial material containing silica in a reactive form, in which themselves, has little or no worth of cement but, in finely grounded and moist state chemically reacted with alkalis (Ca(OH)2) at ordinary temperature to form cementing compounds (Dhinakaran and Gangava, 2016; Naresh and Dadapeer, 2017; (American Society of Testing Materials (ASTM), 2019)

The alternative materials like Pozzolan also referred to as Supplementary Cementing Materials (SCMs) in cement and concrete applications are not just reducing energy consumption and CO_2 emissions but lowered heat of hydration, increased strength and durability, decreased permeability at higher rate and possesses high resistance to sulphate attack (Mertens, *et al.*, 2009; Sandhu and Siddique, 2017). Generally, the use of pozzolanic materials such as rice husk ash, fly ash and microsilica as partial replacement of

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Portland cement to produce concretes and mortars is acceptable (Fapohunda, *et al.*, 2017). Rice husk ash, metakaolin, fly ash and microsilica and Bamboo Leaf Ash (BLA) have been studied and confirmed also as a pozzolanic material (Villar–Cociña, *et al.* 2011; Moraes, *et al.*, 2019) while Palm Fruit Fibre Ash (PFFA) still receives low attention of researchers in the studies of alternative concrete cement materials.

Bamboo is a Gramineae family of plants widely grown in the tropical and slightly temperate regions of the world such as Africa, South America and Asia with above 1% of the total forest area coverage in this regions. The estimated utility values of bamboo were over 20 million tonnes annually as at 2005 based on the usage by many industries like brewing, food, construction, agriculture, textile etc. (Lobovikov, *et al.*, 2007). The bamboo leaf waste generated by these industries are massive, and according to Villar–Cocina et al. (2011), another use of this industrial waste has not been established. They are often burnt in the open air and thereby contributing to environmental pollution, air contamination and carbon footprint while several others where left decaying under the bamboo trees.

Oil palm (*Elaeis guineensis*) is cultivated at a vast scale as a source of oil in West and Central Africa, where it is originated, and in Malaysia, Indonesia, and Thailand. Nigeria among the palm oil producing country with more than 2.5 million hectares under cultivation as at 2011. Today Nigeria is the fifth largest producer with less than 2% of total global market production of 74.08 million tone, more energy is being channeled towards the plantation of oil palm tree among the local investors and farmers in Nigeria to the tune of 37.5m oil palm trees in 27 states (Nigeria Business Report, 2022). Palm oil fruit fibre is part of the oil palm biomass residue periodically left in the field after oil extraction and considers as hazardous material creating environmental problems (Abdul Khalil *et al.*, 2012). A large fraction of the fibre and much of the shell are used as fuel to generate process steam and electricity in the palm processing mill itself resulting in further aggravating the environmental challenges.

The ashes are usually obtained from the calcination of the waste materials at the determined optimum calcined temperature in order to obtain maximum strength for the use in blended cement. As opined by Fapohunda, *et al.* (2017) blended cements are widely acceptable in many part of the word due to its effectiveness to meet most of the requirements of durable concrete. The study of Nehring, *et al.* (2022) affirmed that 30% replacement of PC with BLA in mortar possesses good strength and yielded resistant to acid attack. In the same vain Asha *et al.* (2014) affirmed the suitability of concrete blend of BLA for civil engineering work with low strength and high durability requirement in their study on experimental study on concrete with bamboo leaf ash. Dacuan *et al.* (2021) evaluated the blends of cement using BLA against corrosion on reinforced concrete structures and concluded that 10% BLA replacement improved the permeability of the concrete and prevent chemicals attack.

The study of Rahman *et al.* (2018) on bamboo leaf ash as the stabilizer for soft soil treatment revealed that blended cement concrete with BLA stands as good soil stabilizer with its proved resistance to magnesium sulphate, sodium sulphate and calcium sulphate media. Umor and Adesola, (2015) reveal that BLA is suitable for the production of the blended cement mortar

Dhinakaran and Gangava (2016) study on compressive strength and durability of bamboo leaf ash concrete established that 15% optimum replacement of BLA with cement present a little compromise in strength and durability characteristics.

Several work has been done on the use of palm fruit fibre in various diverse as: production of Hybrid biocomposite (Abdul Khalil *et al.*, 2012); Reinforced gypsum–cement based wall panels (Abuh and Umoh, 2015), replacement for coarse aggregate in light weight concrete (Emiero and Oyedepo, 2012), fired clay brick (Kadir, *et.al.*, 2017) however, research on PFFA as pozzolan material and its blends with concrete is not yet established hence, the motivation for this study which combined the BLA and PFFA to create a synergetic effects due to their chemical composition to achieve a sustainable binder with good strength, durability and unchallenged environmental impacts.

2. MATERIALS & METHODS

Materials

The materials used for this research work includes: Elephant branded Portland lime cement CEM II/A–L, 42.5 N conforming to BS EN 197–1 (2011) and NIS 444–1 (2018), River sand free of debris and organic materials with a minimum particle size of 300 μm in accordance with BS EN 12620 (2013) Crushed granite stone that passed through 13.50mm sieve size and retained on at least 9.50mm sieve size in accordance with ASTM C 33–10 (2003). Bamboo leaf ash and Palm fruit fibre ash and potable water as specified by BS

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EN 1008 (2002) and ASTM C 1602 (2012). A chloride free Complast SP430 super plasticizing admixture made by Purechem manufacturing limited was used to boost the workability of the blended concrete.

Figure 1: Bamboo Leafs

Figure 2: Palm Fruits Fibres

Methods

Bamboo Leafs and Palm Fruits Fibres was calcined through open burning process to reduce the materials bulk content to ashes, the obtained ashes were sieved to be free of particles and impurities before grinded in grinding mill, further calcination in gas fire crucible furnace with attached thermocouple 1300°C under control temperature was carried out at foundry workshop, Technical Support Unit, Faculty of Technology University of Ibadan. The reactivity of the ashes was examined through Modify Chapelle test and X–ray fluorescence (XRF) while the mineralogy of the ashes was determined with X–ray diffraction (XRD) techniques and SEM/EDX techniques was adopted for the morphology of the ashes. BLA/PFFA in 70/30 proportions was incorporated in the blended concrete at 0 to 25% bwob contents at a 5% steps interval, this was denoted as BLAPFFA–5 to BLAPFFA–25 as presented in Table 1 using 0.46 water–to–binder ratio (W/B) with addition of water reducer admixture in proportion of 1.2lts to 50kg cement weight as specified by the producer to achieve the workability of the mixture. The superplasticizer used has a specific gravity of 1.18. The concrete was mixed mechanically, workability test was conducted on the fresh concrete before casting into pre–oiled steel concrete cubes 150mm x 150mm x 150mm, the blended concrete was well compacted on concrete table vibrator. The concrete cubes were demoulded after 24 hours and subjected to water immersion till the testing dates. The cubes were removed after the due dates for compressive strength test in accordance with BS EN 12390–3 (2019).

Table 1: Concrete Mix Composition with BLAPFFA Blended Concrete

Concrete mixes	Material constituent – kg/m3							(W/B)	Additional water
	Water	Binder Composition			Sand	Granite	Superplastiser (1.2lts to 50kg cement weight)		
		PC	BLA	PFFA					
Label	170	370			837	1023	8.88	0.46	
Control		370	0	0	837	1023	8.88	0.46	0
BLAPFFA –5		352	13	6	837	1023	8.88	0.46	0
BLAPFFA –10		333	26	11	837	1023	8.88	0.46	0
BLAPFFA –15		315	39	17	837	1023	8.88	0.46	8.5
BLAPFFA –20		296	52	22	837	1023	8.88	0.46	25.5
BLAPFFA –25		278	65	28	837	1023	8.88	0.46	51

Test methods

Modify Chapelle test

The apparatus was regulated to a temperature of 90°C. 1g of pozzolan was weighed into the dry clean Erlenmeyer where 250 ml of the distilled water was added using a calibrated pipette and about 2g of CaO weighed was added. Continuous stirring started with placement of stirrer in the Erlenmeyer and the tap with the thermometer and the cooling column to homogenise the content of the Erlenmeyer, the apparatus was placed on the heated stirrer to commence heating. Confirmation of temperature of 85°C±5 was performed while the process stir and heat was allowed for 16 hours uninterrupted. The content was cooled to room temperature before 250 ml saccharose solution was added and stir for 15 minutes on the magnetic stirrer after which about 200 ml was filtered out of which 25 ml was taken with a calibrated pipette for titration. The same proceeding was made without pozzolan to serve as control. Titration was done with a burette using the HCl 0,1N solution with phenolphthalein as indicator.

XRF TEST

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The XRF of the binders was conducted on the powdery specimens using the total cement analyser apparatus to determine the material's chemical and oxides compositions as described in Nduka, et.al. (2022).

XRD TEST

XRD (Kα reflections) was performed on finely ground and homogenised BLAPFFA blended concrete samples to observe the samples' crystalline phases and hydration products. The scan range of 4°– 75° with a two–theta steps of 0.026261 at 8.67 seconds per step was adopted. Tube current was 40mA and the tension was 45VA. A Programmable Divergent Slit was used with a 5mm Width Mask and the Gonio Scan was used. As the sample and detector rotate through their respective angles the intensity of diffracted X–rays was constantly recorded.

3. RESULTS & DISCUSSIONS

Physical properties of the constituent's materials of BLAPFFA blended concrete

Figure 3 shows the sieve analysis of the fine and coarse aggregates investigated in this study, Particle Size Distribution (PDS) plot in the figure shows the fine aggregate sample to be medium sand as classified in Shetty's (2004), while uniformly graded stone was used as coarse aggregate for the study. Table 2 revealed the physical characteristics of the sand where Fineness modulus = 2.87; Cu = 5.83; Cc = 0.48; specific gravity = 2.60; water absorption = 1.38% and D60, D30, D10 = 0.7, 0.2, 0.12 respectively. The granite analysed as shown in the table had a specific gravity of 2.65; water absorption of 1.20%; aggregate crushing values of 24.5%; Cu = 1.86; Cc = 1.61. These findings established the efficacy of reducing dust content in coarse aggregate and sieving out finer particles of the river sand with 300 µm sieves in compliance with requirements as in (Aïtcin, 1998).

Table 2: Physical and Mechanical Properties of Aggregates

Properties	Sand	Granite
Bulk density	1667kg/m3	2650kg/m3
Specific gravity	2.6	2.65
Moisture content	6.27%	-
Fineness modulus	2.87	-
Aggregate crushing value	-	24.50
Water absorption, %	1.38	1.2
D_{10}, mm	0.12	7.5
D_{30}, mm	0.2	13
D_{60}, mm	0.7	14
C_u	5.83	1.86
C_c	0.48	1.61

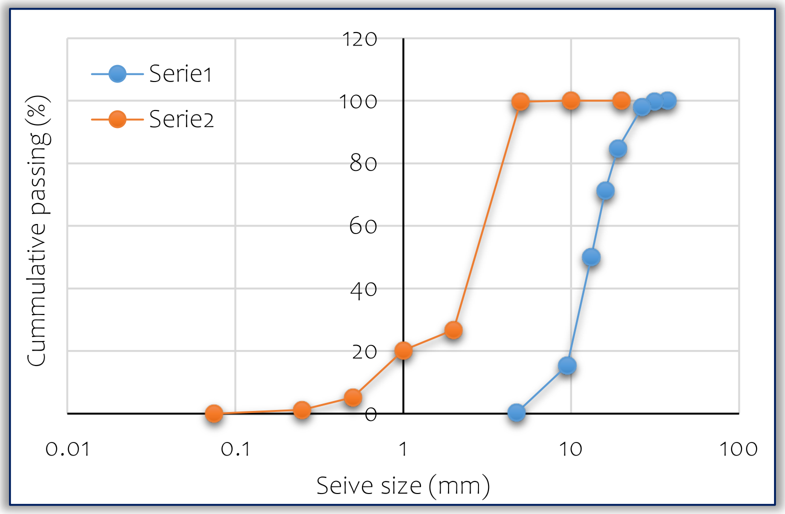

Figure 3: Particle Distribution of Aggregates.

Pozzolanic Reactivity of BLA and PFFA

Table 4 revealed that BLA and PFFA calcined at 700 °C and 800 °C respectively, recorded the reactivity level of 2353.53mg and 1233.3 $Ca(OH)_2$ fixed by pozzolan respectively, this implies that BLA and PFFA possess pozolanic properties and can be selected for PC replacement in concrete as the ashes will consume the hydrated lime in the process of cement hydration.

Table 4: Pozzolanic Reactivity of BLA and PFFA

	BLA	PFFA
Temperature (°C)	700	800
Volume of HCL	2.6	12
Reactivity (mg) $Ca(OH)_2$ fixed by pozzolan	2353.53	1233.3

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Chemical properties of Binders

The oxides composition of PC, BLA and PFFA is presented in Table 5, it can be observed from the table that the results manifested a high silica content of 62 % and 67 % respectively, this is, a measure of the reactivity of BLA and PFFA, silica which according to Kumar and Venugopal, (2013) is identify as the compound in control of the strength in concrete. Silica content recorded for BLA is similar to the results of Nehring, et al., (2022). This further confirmed BLA and PFFA to possess the ability to enhance the strength development process if used in concrete production. Table 5 also revealed that the sum of SiO2+Al2O3 + Fe2O3 for PFFA exceeds 70% while BLA is a little less, this validated the facts that PFFA and BLA are in the same category with the Class F fly ash with high pozzolanic characteristics as stated in ASTM C618 (2008) However, the Table 5 further disclosed an issue of concern that is, the high loss on ignition, which is a measure for the residual carbon content, this may be due to the ashes production processes, hence, did not meet the ASTM C618 (2008) limits for loss on ignition of 6% for class C and F fly ash.

Table 5: Chemical Composition of Binders

Elemental Oxides (%)	PC	BLA	PFFA
SiO2	18.92	62.0	67.0
Al2O3	5.63	2.7	5.0
Fe2O3	3.48	2.7	2.6
CaO	60.74	6.7	3.1
MgO	1.12	1.3	0.6
SO3	0.98	0.4	0.2
Na2O	0.08	0.4	1.8
K2O	0.02	5.2	2.5
TiO2	-	0.2	0.2
P2O5	-	0.6	0.6
Mn2O3	-	0.14	0.07
Cr2O3	-	0.01	0.01
LOI		18.3	16.67
SiO2 + Al2O3 + Fe2O3	28.08	67.4	74.6

Mineralogical phases of BLA and PFFA

Adequate knowledge of an arrangement of components and phases is key to the properties such as strength and durability of concrete. Figure 4 and 5 depicts the phase's structure of BLA and PFFA, underlining the mineral constituents found in BLA and PFFA tested samples. The diffractograms of BLA as presented in figure 4 shows the reflection of quartz (SiO2), Graphite–2H, Lime, syn (Ca O), Osumilite (K – Na – Ca – Mg – Fe – Al – S…), Sodalite (Al6 Na8 (Si O4)6 Cl2), Anhydrite (Ca S O4) and Clinochlore (Mg, Fe +2)5 Al (Si3 Al) O…). Also shown in figure 4 is the highest peak intensity which occurred at 26.93° 2θ with Sodalite and seen to be constantly dominating the peak. Figure 5 shows the diffractograms of PFFA with the reflection of quartz (SiO2), Anhydrite (Ca S O4), Lime, syn (Ca O), Osumilite (K – Na – Ca – Mg – Fe – Al – S…) and Calcite Ca C O3. Calcite has the highest peak intensity which occurred at 27.03° 2θ. The XRD scan terminated at 70° position for BLA and PFFA.

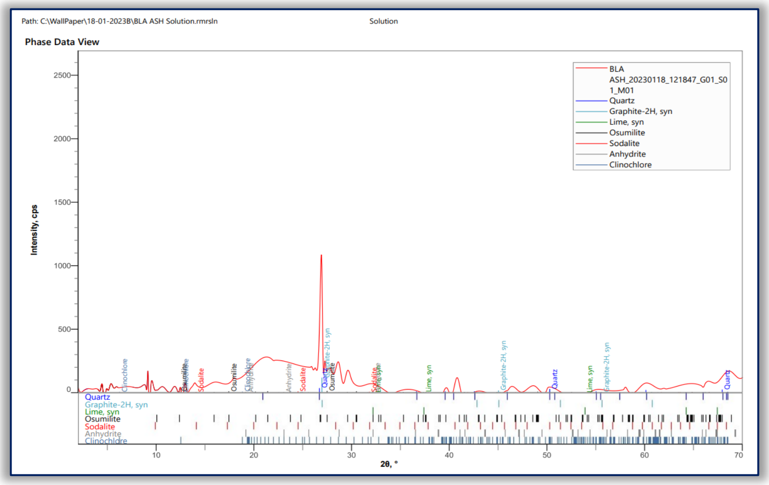

Figure 4: XRD Pattern of BLA

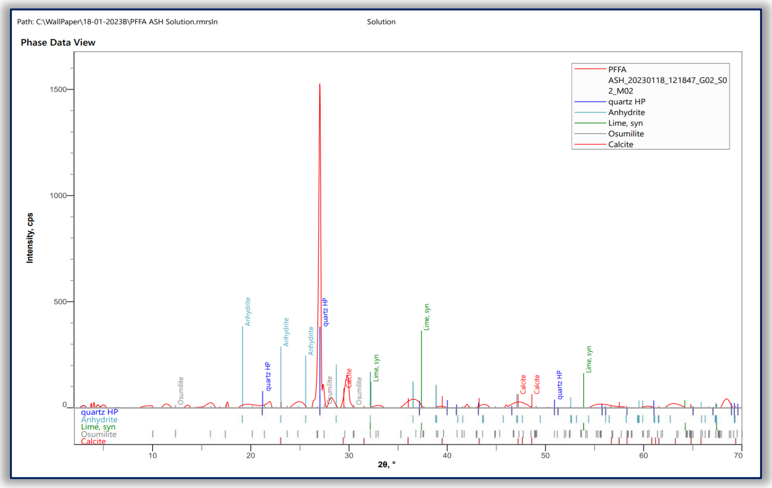

Figure 5: XRD Pattern of PFFA

The quantitative analysis revealed that Graphite–2H mineral takes the most dominant phase with 15%, followed by Sodalite and Clinochlore with 13% by weight in the calcined BLA while the most dominant mineral present in the calcined PFFA is Anhydrite and Calcite. Quartz mineral which was described by Danner, et al. (2018) as invulnerable mineral to heat treatment is presented in BLA and PFFA diffractograms phase spectra at 4%

and 3% respectively. BLA and PFFA diffractograms phase spectra showed that osumilite mineral was present at 2% and 3% in BLA and PFFA respectively. Osumilite mineral possess calcium and silica oxides which was identified as major oxides in binder's chemical oxide composition, hence, present BLA and PFFA as having good potential for cement production Saxena, et al. (2020). The XRD examination confirms the SiO2 chemical oxide composition dominance in the XRF analysis this implied that calcining BLA to 700 °C and PFFA to 800°C can produce an amorphous material suitable for concrete enhancement.

Characterization of BLA and PFFA

Figure 6 and 7 a & b showed the BLA and PFFA SEM micrograph under 600μm magnification and EDX peak results. BLA SEM images in figure 6a depicts flat, needle–like and angular particles denoted as A, B and C respectively. The SEM image also revealed greyish and whitish colour with dark sports between the grains. This result is similar to the findings of Kolawole, *et al*. (2021), Nduka, *et al*. (2022) and Moraes, et al. (2019). PFFA SEM images presented in figure 6a show angular, irregular and more of spherical nature with noticeable clustered form denoted as D, E and F respectively. PFFA SEM image further revealed grey and darker colour sports between the grains. The greyish and whitish colour shown in the images portrait the ashes to be more of reactive substance and they are reactive in this context while the flat shape of BLA may likely make it to required more water for their particle lubrication. In figure 6b and 7b the EDX spectra show silica in high content this silica enrichment in the BLA and PFFA samples are similar to the obtained result in XRF.

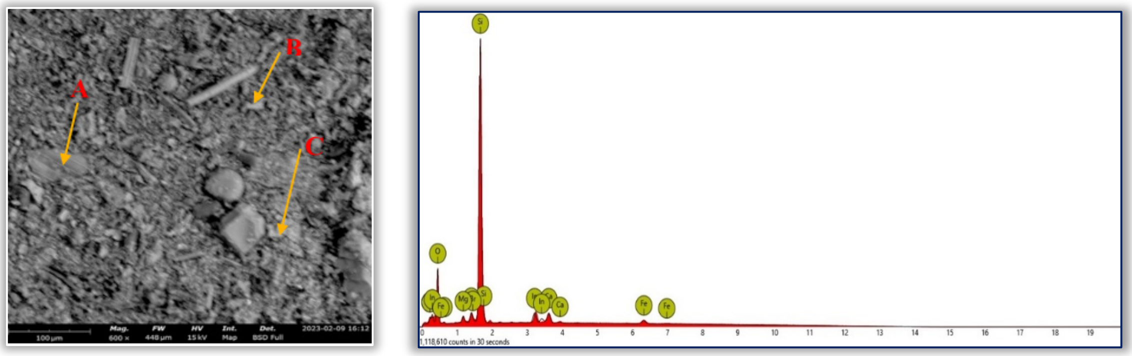

Figure 6a & b: SEM Image of BLA at 600μm Magnification and EDX Microanalysis of BLA

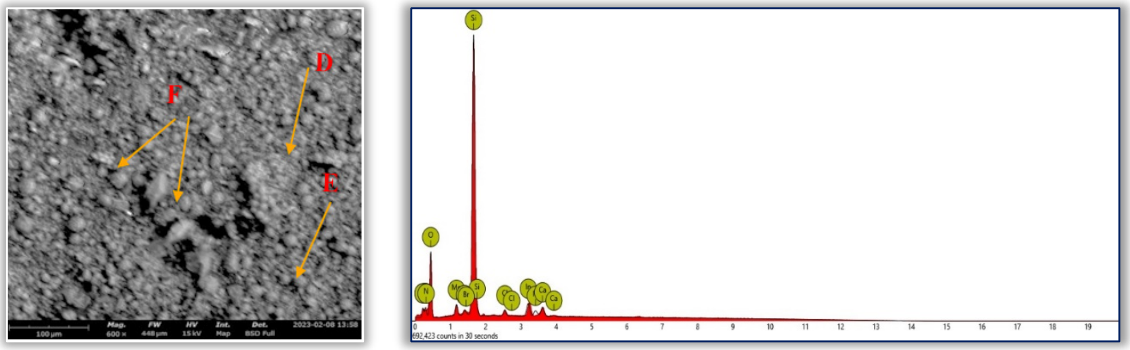

Figure 7a & b: SEM Image of PFFA at 600μm Magnification and EDX Microanalysis of PFFA

Physical and Mechanical Properties of BLAPFFA Blended concrete

Physical properties of BLAPFFA Blended concrete

The physical properties of the PC, BLA and PFFA are shown in Table 6. It can be seen that about 94% of the materials' particles have sizes less than 90μm. The materials were within the limit of 34% fineness set by ASTM C618 (2012). The specific gravity of PC was higher than BLA, and PFFA (3.12, 2.53 and 2.5 respectively).

Table 6: Physical Properties of BLAPFFA Blended Concrete

Parameters	PC	BLA	PFFA
Fineness (% residue on 90 μm sieve)	0	6.33	5.33
Initial setting time (min)	60	–	–
Final setting time (min)	180	–	–
Soundness (mm)	0	0	1
Specific gravity	3.12	2.53	2.5

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It was also observed from the table that the initial setting times of cement is higher than the specified standard of 45 minutes but its final setting time recorded a lower time, which is within the specified standard of 600 minutes BS EN 196–3 (2016).

Setting times of BLAPFFA blended concrete

Figure 8a and 8b shows the setting time of cement paste having pozzolans incorporated at various replacement levels. It was observed that the initial setting times of BLAPFFA blended at various replacement is higher than the specified standard of 45 minutes for PC while its final setting time recorded a lower time, which is within the specified standard of 600 minutes BS EN 196–3 (2016). Figures 6b indicated that BLAPFFA blended concrete at 10% replacement recorded the highest final setting time of 424mimutes, the figures also affirmed that both the initial and final setting times of the paste incorporated with pozzolans increased as the replacement level of the pozzolans increased.

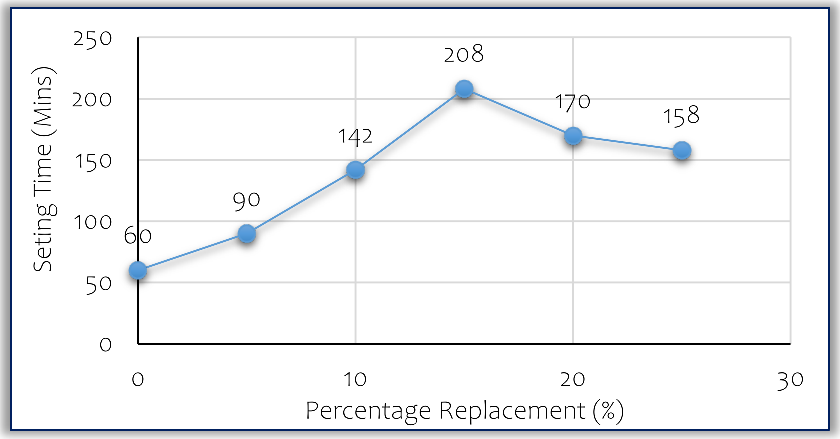

Figure 8a: Initial Setting Time

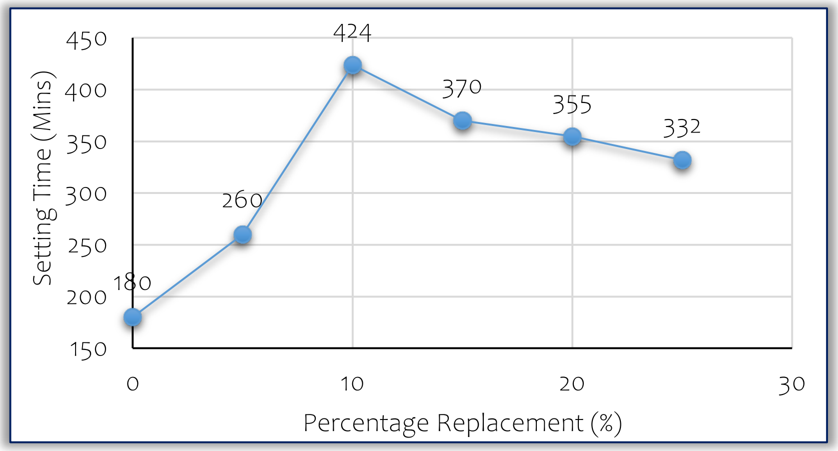

Figure 8b: Final Setting Time

Workability of BLAPFFA blended concrete

Figure 9 showed the variations in the workability of BLAPFFA blended concrete using slump test and the results indicated a lower degree of workability for the blended concrete from control to 25 % replacement level that is, the more the increase in replacement levels for the pozzolans the lower the degree of workability. This observed slump values can be attributed to the high moisture absorption capacity of the pozzolan particles in the mix, also the flat nature of BLA as revealed in its SEM analysis could be responsible for increase in the water demand since its contents is more in the percentage replacement. As reported in Efe, *et al*., (2019) increase in the pozzolans percentage replacement thus caused it to transit from plastic mix to stiff–plastic mix hence, more water request.

Compressive Strength of BLAPFFA blended concrete

Figure 10 shows the results of compressive strength for the samples of blended concrete with BLA and PFFA at 7, 28, 56 and 90 days. The compressive strength increased with the curing age as opined by Voit et al., (2020) that concrete containing pozzolanic materials are known to gain strength slowly at the early days of curing. The strengths were also noticed for decreased with addition of the additive (BLA and PFFA) except at 5% and 10% replacement which at 28days hydration period surpassed the control sample with

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2.54% and 1.62% strength improvement respectively. The highest compressive strength value of 46.58 MPa was obtained at 10% BLA and PFFA replacement at 90 days curing age followed by the control mixture with 45.37 MP and 5% replacement with 44.18 MPa. This implied that incorporation of SCMs through the pozzolanic reaction as opined by Fapohunda, *et al.* (2017) is responsible for the increase in the strength at the later age.

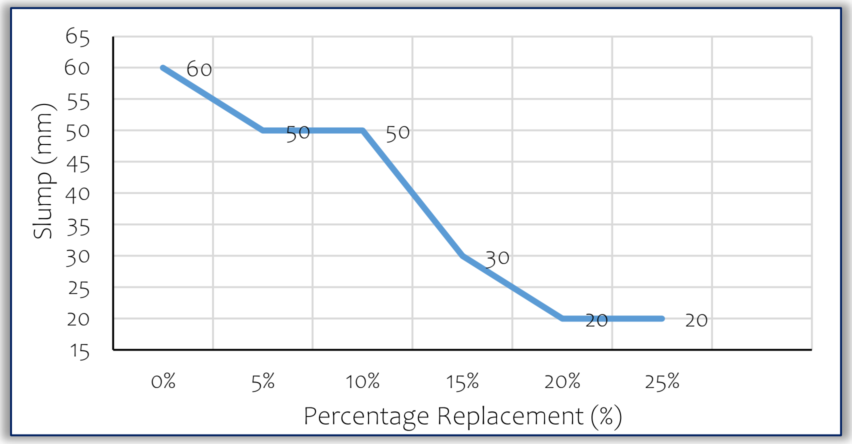

Figure 9: Slump Values of BLAPFFA Blended Concrete

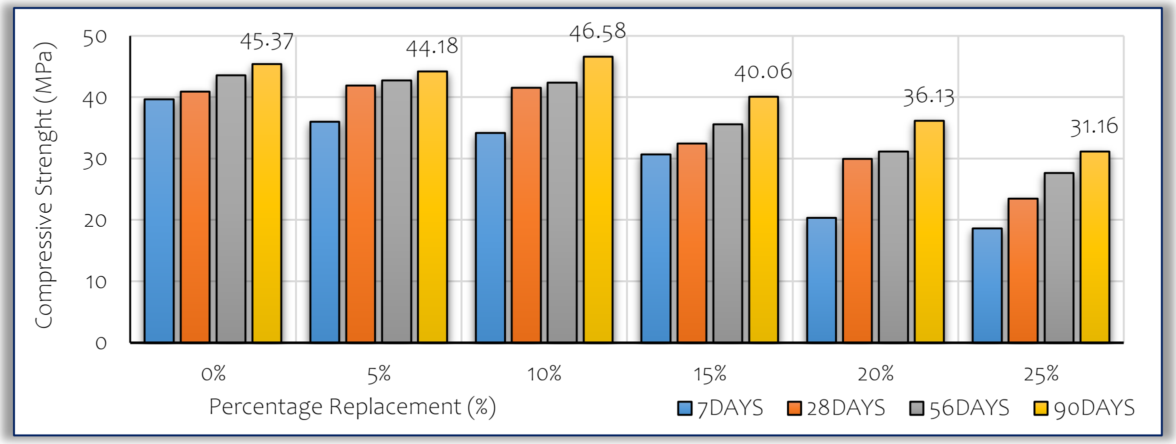

Figure 10: Compressive Strength Development of BLAPFFA Blended Concrete

4. CONCLUSIONS

The following conclusions are drawn from the study:

- BLA and PFFA has a pozolanic property and can be trusted to consume the hydrated lime in the process of cement hydration hence, can be selected for PC replacement in concrete.
- The Silica content of BLA and PFFA is higher than that of PC and the summation of silica oxide ($Si O_2$), alumina oxide (Al_2O_3) and iron oxide (Fe_2O_3) for PFFA is greater than 70% specified by ASTM C–618, 2001 and can complement the BLA that is less than 70%. Thus BLA and PFFA can be adopted as SCMs in concrete production.
- The mineralogical phases of BLA and PFFA both indicated quartz ($Si O_2$), Osumilite (K – Na – Ca – Mg – Fe – Al – S…) and Anhydrite ($Ca S O_4$) which possess essential oxides that are required in binding substance in this context.
- BLA and PFFA SEM/EDX images and spectra established them as a reactive substance with higher silica contents and so, suitable for concrete improvement.
- The compressive strength of BLAPFFA blended concrete increased with the curing age and the optimum replacement of PC with BLA and PFFA is 10% as this replacement level gave a higher compressive strength when compared with the control sample.

This research will be further studied the morphology and durability of the BLAPFFA blended concrete for sustainable construction.

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