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Research article

Development of sustainable interlocking concrete paving blocks using bamboo leaf ash and metakaolin

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ABSTRACT

Eco-friendly interlocking concrete pavement block was developed using the admixture of bamboo leaf ash and metakaolin. This was done to develop an eco-friendly interlocking paving block for sustainable pavement construction. Bamboo leaf ash and metakaolin were added as a supplementary cementitious material. The supplementary cementitious material (bamboo leaf ash and metakaolin) were admixed and added at 0, 5, 10, 15, 20, 25 and 30 % replacement of cement. The workability of the fresh concrete (slump) at the varied percentage additions, and the mechanical properties of the concrete at 7, 14, 28, and 56 days of curing were assessed, including their microstructural characteristics. The outcome of the research showed that increasing the percentage of (bamboo leaf ash + metakaolin) reduces the workability of the concrete. With a 20 % addition of this cementitious material, the developed fresh concrete became unworkable. In addition, replacing up to 10 % of the concrete in the pavement with bamboo leaf ash and metakaolin increased the mechanical strength of the concrete by 28.7 %. At 30 % a percentage increase of 3.6 % was recorded. However, the strength at 5 % was still adequate for pavement construction with a 13.62 % increase in mechanical strength. The compressive strength at 5 % and 10 % addition of the supplementary cementitious material at maturity met the criteria for constructing a semi-rigid pavement, using IRC standards. The microstructural assessment showed that the number of pores in the mature concrete samples decreased at 10 % addition of bamboo leaf ash and metakaolin. The research data provides construction workers, researchers, and highway engineers with vital information regarding the viability of these sustainable materials for pavement improvement.

1. Introduction

The first interlocking paving stones appeared in Netherlands in the middle of the 19th century and served as an alternative to bricks. Due to scarcity, bricks became increasingly difficult to obtain after the 2nd World War. focus shifted to the production of

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concrete paving blocks. Since then, there have been advances in improving their physical and mechanical properties, durability and overall strength. Nowadays, researchers consider the addition of industrial and agricultural wastes that exist in abundance in society as a partial replacement of the concrete materials used in these paving stones to improve their properties [1–5]. This research, motivated by the need to find a sustainable use for abundant agricultural waste (agro-waste) produced in society are now important. Due to increased industrialization in all spheres of life, current projections show that industrial and agricultural wastes produced in society will reach 27 billion tons in 2050 [6]. This gives indications that there will be a continual increase in the volume and quantity of waste produced from industrial, agricultural, and mining activities and improper disposal of these wastes could lead to the release of harmful compounds such as silica into the society. Thus, the need for effective management, disposal, or re-use of these wastes.

Agricultural wastes thrown away or burnt in fields can have detrimental consequences on the environment especially when a compound such as silica leaks onto the water table. These agricultural waste products when channeled appropriately serves as an additive and a sustainable alternative to concrete [7–9]. One of these wastes is leaf from bamboo: a type of plant categorized in a grass group called the *Poaceae* (*Gramineae*) family [10]. Currently, there are over 1200 species of bamboo found in the world [11], implying that there is abundant waste product generated when its harvested and used for various purposes. For example, waste generated from bamboo leaves could be repurposed for construction use as 95 % of the waste generated when burnt contains amorphous silica (SiO_2), which could increase concrete durability and decrease the susceptibility of the cement matrix to acid attack [12–15]. Aside from SiO_2, other compounds found in the agro-wastes such as CaO, Al_2O_3, and Fe_2O_3 can also improve the strength and durability of concrete mixes [16–33]. Previous research depicts the recent developments on the application of (agro-waste) sugarcane bagasse ash and limestone in eco-friendly geopolymer concrete [34–37]. Another research focused on the application of Cu slag as a fine aggregate in concrete [38]. Alahmari et al. [39] conducted a review of recent developments regarding the durability performance of eco-friendly geopolymer concrete. Other research involving the application of agro-wastes as partial replacements in concrete have been studied with important results worthy of note for further extensive experimentation [40–44].

It's important to note that another material that exists in abundance globally is metakaolin (MK): an anhydrous compound that naturally exists in clay but is produced in abundance from waste paper sludge and serves as geopolymers when used in concretes. It has shown promising results in improving the properties of concrete and researchers have extensively studied them along with other materials [23,24]. Previously [45,46], the combined effect of MK and silica fume in the strength properties of concrete was studied. In addition, references [47,48] evaluated the consistency of concrete incorporating fly ash and MK and the results have been positive. Thus, it has been established that the addition of MK, silica fume and/or fly ash improved concrete durability. However, these researches did not assess the pavement application of these developed concrete. This research attempts to fill this gap by evaluating the influence of adding silica (i.e., ashes generated from agro-waste such as bamboo leaf ash) in the production of an eco-friendly paving block for pavement construction. With the world mostly relying on road transportation, there is a need for adequate pavement: rigid, semi-rigid, or flexible conditions requiring minimal maintenance. As a result, studies have focused on improving these pavements, especially the flexible and rigid types. However, there is limited information on improving the structural quality of semi-rigid pavement, which involves the use of paving blocks. As a preferred pavement, they are currently gaining popularity, due to their durability especially when used on soils with low bearing capacity.

In recent years, the construction of these pavements has increasingly utilized innovative auxiliary materials as alternatives to cement. As a result, researchers have recently begun to focus on using interlocking pavement blocks in road construction due to their benefits of outperforming both asphalt and regular 'poured-in-place' concrete. These benefits include resistance to degradation caused by freeze-unfreeze cycles and de-icing salts. In addition, this distinctive pavement has aesthetic and durability properties, which makes it a viable alternative in pavement construction. The study brings into play the utilization of waste to improve the cementitious properties of the concrete material used in the construction of these pavements. In addition to promoting sustainability, using these wastes as partial replacements for cement could lower the overall cost of concrete production. Studies have shown that utilizing these wastes reduces the overall cost of producing concrete. Furthermore, using agricultural waste in concrete enhances its strength and durability properties. Therefore, the study adopted the usage of agricultural waste (bamboo leaf ash), a natural amorphous pozzolanic material rich in silicon dioxide (SiO_2), and a geopolymer (metakaolin) as a green alternative for cement. Previous research focused on using bamboo leaf ash (BLA) or bamboo straw ash (agricultural waste) in concrete production. However, this research focused on the synergetic effect of using agricultural waste and a geopolymer in concrete production and the application in pavement.

2. Materials and methodology

2.1. Materials source

The materials (fine aggregate, and cement) used in the study were sourced locally in Nigeria. Fine aggregates with a 4.7 mm nominal size are found naturally in Nigeria and met NIS 13:1974 standard [49] as well as AASHTO M 6 [50], and AASHTO M 80 [51] specifications. The choice of using locally available fine aggregate was to aid a durable yet cost-effective mixture. The authors obtained BLA locally in Nigeria and used a portable water source nearby. MK – a component for the study locally existing in Oye, Ekiti, Ekiti State, Nigeria, thus locally available MK was used for the study.

2.2. Sample preparation

De-hydroxylation of kaolinitic clay: a phenomenon influenced by particle size and crystallinity was performed in a furnace. The results indicated that at 750^{o} C, de-hydroxylation of the kaolinitic clay occurred, hence was the standard temperature used throughout

Table 1
Change in weight during de-hydroxylation.

Temperature (°C)	SAMPLE A (kg)	SAMPLE B (kg)	SAMPLE C (kg)
300	48	47.5	47.8
400	42	41.8	42.2
500	38	37.5	38.1
600	34	33.7	33.9
650	33.5	33.2	33.6
700	33	32.9	33.5
750	32.8	32.7	33.2

Table 2
ASTM requirement for pozzolanic material.

S/N	Constituents	ASTM C618	MK	BLA
1	$SiO_2 + Al_2O_3 + Fe_2O_3$	Min. of 70	72.4	70.9
2	SO_3	Max. of 4	3.2	2.9
3	Moisture content	Max. of 3	2.1	2.05
4	Alkalis (Nao and K_2O)	Max. of 1.5	1.1	1.21
5	Loss on ignition (LOI)	Max. of 10	5.8	5.7

Source: ASTM C618-12, 1994

Fig. 1. Admixture of MK and BLA powder (BLA + MK).

Table 3
Weight value of the Constituents.

Mix Design Proportions (per m^3)		
Constituents	Quantity	Unit
Coarse aggregate	500	kg
Fine aggregate	250	kg
Cement	192	kg
Bamboo Leaf Ash + Metakaolin	0–57.6	kg
Water	96	L

the study. Table 1 shows the results of measuring the weight of kaolinitic clay before and after de-hydroxylation. In preparation for the bamboo ashes, bamboo leaves were sun-dried and burned under a regulated combustion process at 600 °C in a furnace. Ashes were obtained from the combustion process using a 45 mm sieve to attain a fineness of not more than 63 mm, as recommended by Ref. [23]. The BLA was sun-dried and then burned for 2 h at 600 °C in a furnace as suggested by Ref. [45]. Table 2 shows the BLA and MK 's pozzolanic characteristics compared with ASTM specification. Fig. 1 shows the addition of MK and BLA (BLA + MK).

Table 4
Concrete mix design.

Percentage Replacement (%)	Cement	BLA	MK	Sand	Granite	Water
0	192	0	0	250	500	96
5	182.4	4.8	4.8	250	500	96
10	172.8	9.6	9.6	250	500	96
15	163.2	14.4	14.4	250	500	96
20	153.6	19.2	19.2	250	500	96
25	144	24	24	250	500	96
30	134.4	28.8	28.8	250	500	96

2.3. Concrete mix design

The research project was performed at the Department of Civil Engineering, Covenant University, Ota, Ogun State, Nigeria. The Maximum Aggregate Size (MSA) determines the minimum amount of cement for concrete pavement. However, for the study and due to the MSA of 19 mm, a small amount of cement was used. Using the weight technique, cement, sand, and granite were batched together. Tables 3 and 4 show the constituent percentages by weight. The study used a ratio of 1:1.2:2.4 for the mix design (see Table 4). Superplasticizers or any other additive that reduces water was not employed thus the water-cement ratio was 0.5. Based on previous research [6], a mean strength of 30 MPa at maturity (28 days) and beyond (Table 3 shows the mix-design) was attained. BLA + MK was substituted for cement at a rate of 0 %, 5 %, 10 %, 15 %, 20 %, 25 % and 30 %, and mixed with cement, fine aggregate and coarse aggregate manually. Interlocking plastic moulds used for casting paving blocks were lubricated to ensure easy removal of samples after the hardening process. In the study, concrete was compacted manually with twenty-five blows from a tampering rod following the recommendation of [6] and a slump test was done using BS EN 12350–2:2009 [46] to verify the workability of the freshly produced concrete.

2.4. Assessment of the fresh concrete property

The workability of concrete was determined by BS 2001 part 102: 1993 [52,53] and ASTM 143-90a [54] specifications. The consistency and ease of mixing and placing the fresh concrete mix was also examined. Concrete was poured into the cone and elevated vertically for 3 s to determine the slump value of each batch.

2.5. Assessment of the mechanical properties of the concrete

After the slump test, the fresh concrete was placed in the lubricated moulds and damped for about 24 h with a polythene covering followed by mould removal and drying of samples in the open. At the daily temperature of southwestern Nigeria, which does not surpass 30 °C, H_2O was sprayed twice each day to enhance concrete strength development. Afterwards, the mechanical properties of the cured concrete samples in the laboratory were examined by following the requirements of BS 2009 [53] after curing for 7, 14, 28, and 56 days. Mechanical test conducted on the samples includes the compressive strength and split tensile strength test at the designated age. The results obtained were compared with the IRC code for interlocking paving blocks.

2.6. H_2O absorption

The absorption capacity of the concrete was assessed by completely immersing the sample in H_2O for 28 days; weight was calculated and compared with the control sample. Following the recommendation of [6], these samples were then oven-dried at a temperature of around 1000 °C to determine any changes in sample masses.

2.7. Linear regression method

The study used linear regression approaches to understand the relationship between concrete strength, the age of concrete, BLA, MK and cement quantities. Equation (1) gives the functional form of the linear regression model used.

$$Y_i = \beta_0 + \sum_{i=1}^{N} \beta_i X_{ik} \tag{1}$$

Where Y_i is the response variable i.e., concrete strength.

X_{ik} is the dependent variable i.e., age of concrete, BLA, MK and cement.

β_0 is the intercept.

β_i represents the parameters for each independent variable.

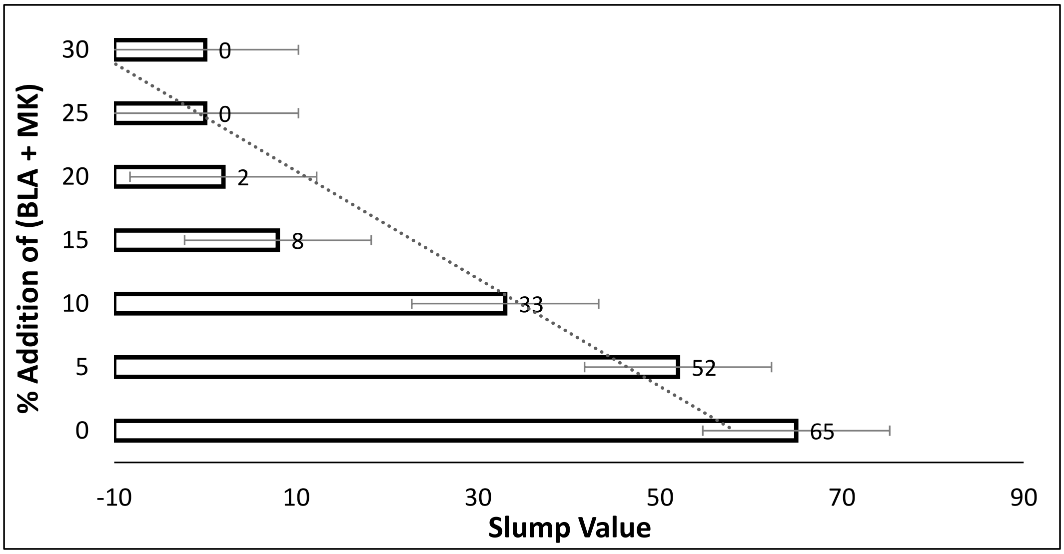


Fig. 2. Slump Test of the concrete.

Table 5
Particleometry of the cementitious materials.

Material & Property	BLA		MK		CEMENT	
	Median	Average	Median	Average	Median	Average
Circle equivalent diameter (μm)	69.4	68.5	77.4	73.1	78.4	73.1
Major axis (μm)	83.9	86.5	88.6	94.2	86.6	92.2
Minor axis (μm)	55.3	54.6	57.8	60.7	53.3	59.8
Circumference(μm)	89	98	431	444	424	441
Convex hull (μm)	301.2	298.7	317	299	314	297
Circumscribed circle diameter (μm)	132.9	121.9	122	119	119	116
Area ($μm^2$)	4.90×10^2	4.81×10^3	5.11×10^3	4.88×10^3	4.84×10^3	4.68×10^3
Volume by area ($μm^3$)	2.33×10^3	2.12×10^3	2.61×10^5	2.71×10^5	2.53×10^5	2.69×10^5
Pixel count	10393	10194	11391	11009	11374	11007
Aspect ratio	0.686	0.606	0.792	0.685	0.786	0.673
Circularity	0.411	0.408	0.293	0.362	0.283	0.326
Convexity	0.813	0.906	0.691	0.729	0.676	0.718
Elongation	0.403	0.394	0.218	0.332	0.208	0.327
Grayscale	178	178	138	136	136	135

2.8. Scanning electron microscopy

Microstructural properties of matured concrete modified with BLA + MK were examined and compared with SEM images obtained with pure concrete at mag. x500 and x1000 using PhenomWorld scanning electron microscope at Covenant University Central Instrumentation Research Facility, Ota, Ogun State, Nigeria.

3. Results and discussion

3.1. Workability properties of BLA + MK concrete

The slump test was used to establish the workability of the fresh concrete considering the addition of BLA + MK at varying proportions of cement content from 0 %, 5 %, 10 %, 15 %, 20 %, 25 % and 30 %. Fig. 2 shows the result of the slump test. It was realized that as the proportion of BLA + MK increases, the concrete workability decreases. This may be because the H_2O-cement ratio used was constant at 0.5. When the proportion of BLA + MK added to the concrete mix was 20 %, the concrete became non-workable, i.e. the concrete became more rigid, and no slump values were recorded. This shows that BLA + MK addition of less than 20 % of the cement was the optimal proportion for the concrete to be workable. This was linked to the absorptive capabilities of the cellulose particle in the BLA, which affects the mixture's specific surface area. Thus, it can be inferred that fine BLA + MK absorbs a substantial amount of H_2O on its surface and stores it in its pores, resulting in a drop in free water and a drop-in slump value. In addition, due to the presence of macro and mesopores within BLA particles, the specific surface area increases as particle fineness increases. This induced greater

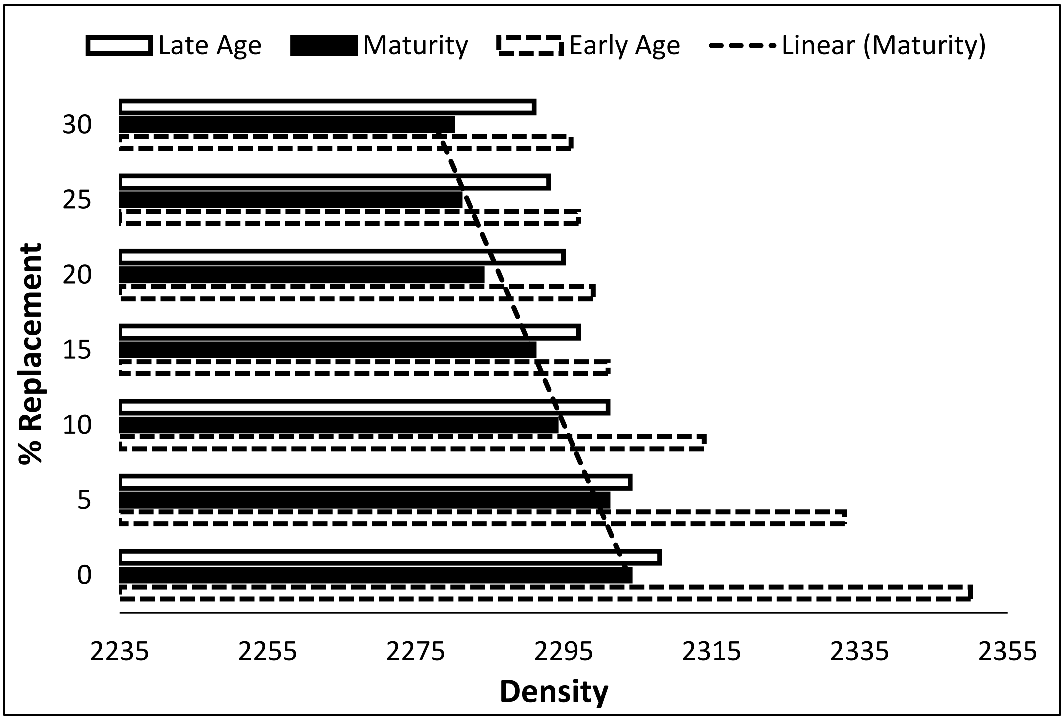


Fig. 3. Density of BLA + MK Concrete across the age of maturity.

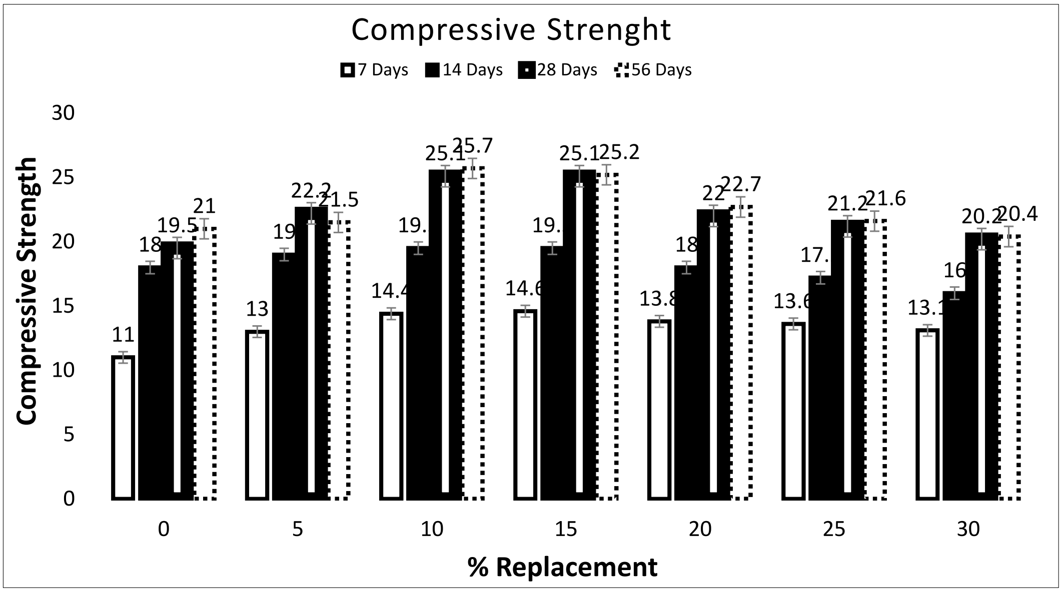


Fig. 4. Compressive Strength of BLA + MK concrete across the age of maturity.

reactivity of BLA and can also reduce the flow of concrete. BLA + MK enhanced surface area may be due to the high carbon content which is primarily responsible for the rigidity of concrete [55]. The addition of MK increased the yield stress and the thixotropic impedance of the cement paste. It is believed that MK can reduce the pores in mortar, thereby refining the pore diameter inside the mortar. Besides, MK can also improve the mechanical properties of the concrete at the initial stage [45–48].

The findings from the slump test are in alignment with previous research on the slump value of concrete using supplementary cementitious materials [51]. The authors suggest that concrete's mechanical strength becomes altered at higher particle size and dimensions resulting in an incomplete hydration process. Additionally, it will have a negative impact on the strength of the concrete

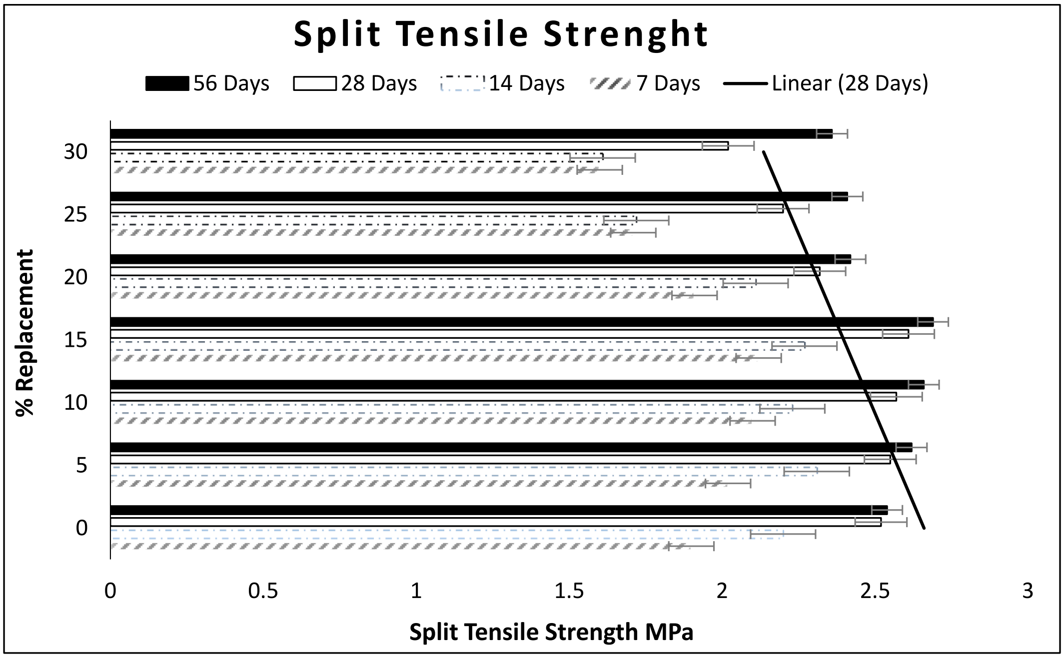


Fig. 5. Split Tensile Strength of BLA + MK concrete across age of maturity.

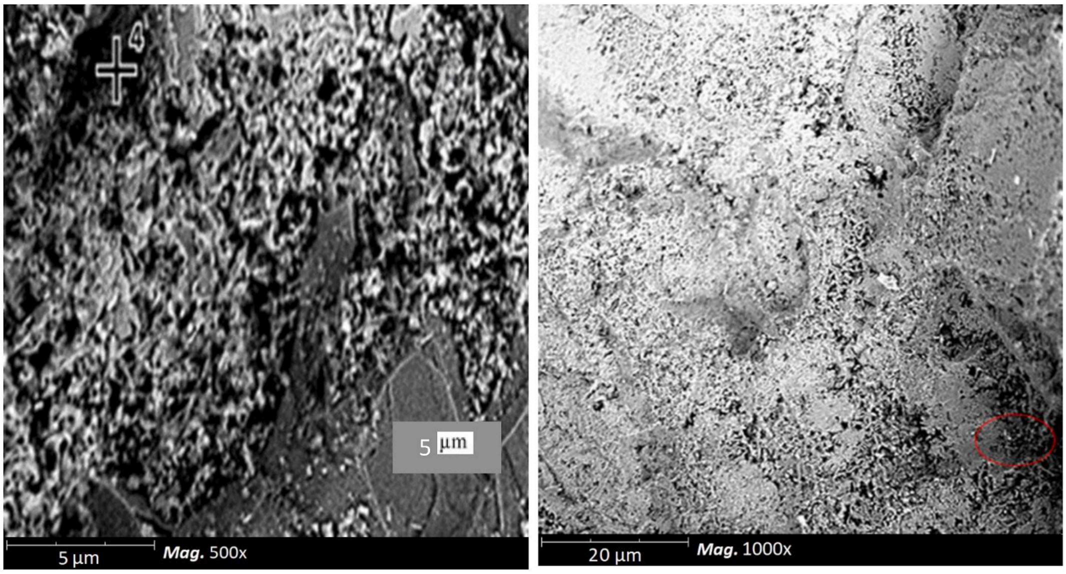
[56]. However, according to particleometry, the cement and cementitious material (BLA + MK) have particle sizes within the range stated in Table 5. This indicates that the hydration process will be complete, and particle size will not affect the strength but will positively affect the concrete's setting time [54]. Moreover, there is a notable connection between cement particle surface area and strength and hydration development implying that the fineness of the cement particles and the supplementary cementitious material will affect the pace of hydration.

3.2. Density of BLA + MK concrete

The outcome of the research revealed that the densities of concretes varied with the amounts of BLA + MK introduced. As shown in the graph (Fig. 2). The density of concrete may impact its strength, additionally, it may reduce the risk of thermal cracking and space savings, hence the test. The density of the concrete with BLA + MK decreases as the curing time increases. This was linked to the effect of ageing of $Ca(OH)_2$ produced during cement hydration on pozzolanic reaction, though $Ca(OH)_2$ must be old enough before the pozzolanic reaction can take place [55]. However, during the second stage of hydration, it was expected that the production of CaH_2O_4Si from the consumption of $Ca(OH)_2$ which has a lower density than the cement components from the initial hydration. Hence, the reduction in concrete density at maturity (see Fig. 3). This finding is consistent with the study by other authors [56–75], that with ageing and maturity concrete density reduces.

3.3. Compressive and split tensile strength of BLA + MK concrete

The investigation of the compressive strength of concretes showed that replacing up to 10 % of the cement in the paving block with BLA + MK increased its strength (see Figs. 4 and 5). There was a decrease in the concrete compressive strength with the addition of BLA + MK at a 5 % increase rate. However, interlocking paving stones for low-traffic areas can still make use of these values. This was attributed to the increase in the compressive strength at 10 % addition of BLA + MK and decrease in the pore size in the cement and MK. One of the extra cementitious elements may be responsible for the improved compressive strength. This followed a similar trend with the research of [56,59–63]. In addition, MK has a high pozzolanic reactivity that allows it to react with $Ca(OH)_2$ released during Portland cement hydration and serves as a micro-filler action that enhances the packing of the cement matrix. In addition, the presence of silica oxides and a significant amount of calcium oxide in the BLA can also improve the mechanical strength of the produced concrete with a 10 % addition of BLA + MK. Similarly, the development of tricalcium silicate and dicalcium silicate is a direct result of the presence of these oxides. In addition, the results at both 10 % and 15 % replacement met the minimum compressive strength as specified by the Nigerian Building and Road Research Institute of Nigeria for interlocking paving blocks. When the Ca_2O_4Si hydrates, it encapsulates agro-waste fibers and improves the cementing properties of concrete (C–S–H). Therefore, this research investigates the microstructural properties of these concrete to have a better understanding of the bonding between the concrete materials.



a: Microstructure at 0% BLA + MK **b:** Microstructure at 10% BLA + MK

Fig. 6. **a:** Microstructure at 0 % BLA + MK, **b:** Microstructure at 10 % BLA + MK.

3.4. Microstructural analysis of BLA + MK concrete

The microstructural properties of matured concrete modified with BLA + MK were examined and compared with SEM images obtained with pure concrete at mag. x500 and x1000. Many researchers have proposed different SEM image analysis methods to study concrete microstructure [21–23]. Fig. 6a and b shows the microstructure of matured concrete without BLA and MK content. It was observed that a tightly packed interface indicates a dense surface at 0 % BLA + MK concrete mix with no additional cementitious material but there were minute pores present. Following a 10 % addition of BLA + MK, the number of pores in the mature concrete samples decreased. This indicates that after hardening, the concrete will grow stronger. Even though the images suggest that the mixtures have adequate matrix-aggregate adhesion, the presence of holes at a high BLA + MK greater than 10 % content will result in a poor interfacial zone.

The implication of these findings suggests a good application of concrete with 10 % BLA + MK in roadway surfacing. The result of the compression test demonstrates that 28 days of curing the concrete resulted in the development of strength enough for pavement purposes and reinforces the possible application of these types of concrete in road surfacing. It was determined that concretes using BLA + MK at 10 % yielded a total strength of 31.3 kN/m^2 at 28 days. This is greater than the 20–30 kN/m^2 recommended by IRC SP 063 for interlocking pavement. It is worth noting that the compressive strength at 15 % addition of BLA + MK at 28 days also meets IRC specification for pavement blocks. At these percentage additions, the compressive strengths are adequate for road construction but fall short of the value specified by the FAA and AASHTO for airport pavement and pavement designed for high-axle vehicles. However, the study showed that none of the batch samples could reach this compressive strength between 7 and 21 days and future studies should focus on evaluating the strength properties of these concrete at older ages. BLA forms CaH_2O_4Si when it reacts $Ca(OH)_2$ [76,77]. According to the authors BLA enhances/improves the compressive strength of the concrete. Thus, authors concluded that addition BLA + MK up to 20 % enhances the compressive strength and workability of concrete. This is due to the ash content of BLA consisting of inorganic minerals, primarily SiO_2, C and K. Mn and Mg are two other common minerals.

3.5. Modelling relationship of concrete strength, age of concrete, BLA + MK and cement contents

The analysis explored the mathematical relationship between the strength of concrete, the age of the concrete, BLA + MK and cement content using linear regression. Equation (2) showed the mathematical relationship between the strength of concrete, age of the concrete, BLA + MK and cement content as well as the model parameters. The model F-value suggests that the model is statistically significant. Model terms are considered significant when "Prob > F″ values are less than 0.0500. Here, the model terms A, B, and A^2 are crucial. The adjusted R-squared value of 0.8441 for the model is fairly close to the predicted value of 0.8045.

$$\text{Compressive Strength} = +51.23A + 0.0213B - 194.5C - 2.007A^2 - 3.18B^2 + 230.83C^2 - 1.21AB - 0.33AC + 0.07BC \quad (2)$$

Where:

A = Concrete Age.

Table 6
ANOVA for response surface quadratic model.

Parameters	The sum of Squares	DF	Mean	F	Prob > F
Model	3410.712	9	378.9681	32.88178	<0.0001
A	1462.498	1	1462.498	126.8961	<0.0001
B	232.0671	1	232.0671	20.13568	<0.0001
C	0.188915	1	0.188915	0.016392	0.8987
A^2	635.0223	1	635.0223	55.09875	<0.0001
B^2	4.964123	1	4.964123	0.43072	0.5151
C^2	9.158432	1	9.158432	0.794646	0.3775
AB	224.758	1	224.758	19.5015	<0.0001
AC	25.87625	1	25.87625	2.245195	0.1412
BC	0.722796	1	0.722796	0.062715	0.8034
Residual	507.1074	44	11.52517		
Cor Total	3917.82	53			

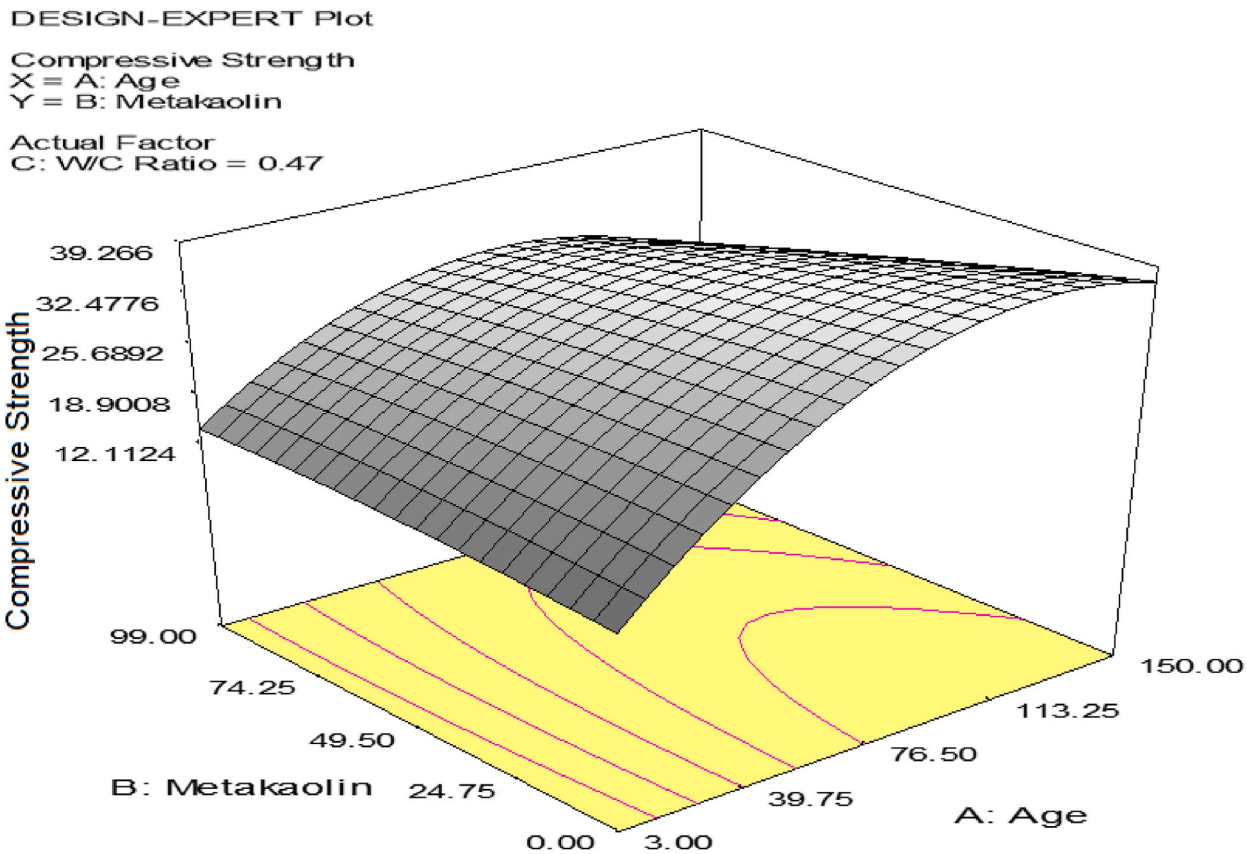


Fig. 7. 3-Dimensional representation of the effect of BLA + MK and concrete age compressing strength of concretes.

B = BLA + MK.

C = Cement content.

The analysis of the model parameters is shown in Table 6. The F-value from the ANOVA table for the model was 32.85 at a statistical significance less than 0.0500. Other significant terms in the model are A, B, and A^2. The significant terms identified show that the concrete age, the proportion of BLA + MK, and the cement as well as the interactions between these components of the concrete play a more significant role in determining the compressive strengths of concretes with BLA and MK. Using a three-dimensional approach, the study further described the relationship between concrete age and MK and concrete compressive strength while holding the water-cement ratio constant (See Fig. 7). From Fig. 7, as the concrete ages from 7 to 56 days, the compressive strength at low MK concentrations rises from 12.1 kN/mm^2 to 37.3 kN/mm^2. Similar behavior was seen at high MK concentrations of 99 Kg. However, the compressive strength of the concrete was lower at a lower age, MK and BLA.

4. Conclusion

The combined admixture of bamboo leaf ash and metakaolin in the development of an eco-friendly paving block was studied. Results obtained show that increase in the percentage of BLA + MK reduces the workability of the concrete. However, at 20 % addition (or more) of BLA + MK, the workability drops and the concrete becomes rigid. Replacement of up to 10 % of the concrete with BLA + MK increased its mechanical strength by 13.62 %. This value is determined to be the optimal addition for BLA + MK in the concrete without any detrimental effect on concrete properties. This observation is due to decrease in the number of pores in the mature concrete samples decreased and the presence of a high percentage of SiO_2 and CaO in the bamboo leaf ash.

Declarations

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Funding

N/A.

Availability of data and materials

Data will be made available on request.
No data associated with this study been deposited into a publicly available repository?

CRediT authorship contribution statement

Ayobami Adebola Busari: Writing – review & editing, Writing – original draft, Supervision, Conceptualization. **Roland Tolulope Loto:** Writing – review & editing, Writing – original draft, Supervision, Formal analysis, Conceptualization. **Samuel Ajayi:** Validation, Methodology, Investigation, Formal analysis. **Seun Daniel Oluwajana:** Methodology, Investigation, Formal analysis. **Ajiboye Eletu:** Validation, Methodology, Investigation, Formal analysis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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