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Potentials of Balanite Endocarp Pod Ash as a Cement Replacement Material

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ABSTRACT

The exponential growth of agricultural wastes such as Balanite seed pod has resulted in waste management issues and finding alternatives through waste recycling is an interesting area of research. Balanite seed pods were collected in Yobe state, washed, dried, crushed. Balanite endocarp pod ash (BEPA) was calcined at 600 °C for 3 hours in a muffle furnace and the biomass was characterized with X-ray fluorescence spectrometer to determine its chemical composition. The analysis showed that the ash composed of silica, alumina and ferric oxide (74.24 wt.%) indicated a strong pozzolan based on American Standard. The BEPA possessed a high loss on ignition of 8.24 wt.% owing to the presence of unburnt carbon in the ash. The ash obtained was sieved with 75 µm sieve and cement was replaced with ash between 2.5 wt.% ~ 12.5 wt.% at interval of 2.5 wt.% to assess its impact on cement properties such as setting time, consistence, mortar strength and water absorption. Results showed that as percentage replacement increases, the water absorption increased while the mortar strength diminished whereas as the curing days progressed its strength improved despite clinker diminution due to cement hydration. 60 days strength for below 10 wt.% BEPA cement blend produced enhanced strength compared with control which is evidence of slow pozzolanic reactions. The optimum percentage replacement with BEPA was obtained at 7.5 wt.% replacement beyond which significantly affected the cement properties especially its strength. It could be agreed that BEPA has potentials to be considered and employed as a cement replacement material.

1. Introduction

Balanite *Aegyptiaca* (laloub) is a spiny semi- evergreen shrub from the family tree of the *Zygophyllaceae* spanning about 12 m high producing yellow date-like

fruits^[1]. Balanite *Aegyptiaca* is also referred to as desert date, fondly known as Laloub in Arabic, Aduwa in Hausa, Hingota in Hindi and Zagum Mitzi in Hebrew. The fruit yield of a mature tree bears as much as 10,000 fruits per annum. A single fruit comprises of (5%-9%) epicarp,

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(28%-33%) mesocarp or pulp, (49%-54%) endocarp, and (8%-12%) kernel with oil content of *B. Aegyptiaca* seed approaches 50% [2-4]. The fruit is a plum like drupe, pubescent when green becoming yellowish and glabrous after ripening [5]. The fruit possesses four main parts namely the epicarp, mesocarp, endocarp and the kernel. The fruit when ripen is a drupe possessing brownish to reddish thin epicarp, a dark brown, fleshy mesocarp and a thick endocarp with the seed [6-8]. The whole fruit yields half of the woody shells which are hard, dense and highly combustible. The Balanite fruits is considered a resilient evergreen tree beneficial to mankind for centuries as well as one of the oldest fruits which dates back to the era of Pharaoh's tombs around the 12th dynasty in ancient Egypt. It is an underutilized tree which possesses numerous uses and its surprising that it has been exploited beyond its potential use. The tree provides many uses ranging from feedstock for livestock to medicinal purposes. They make good fuel as well as good charcoal and particle board [9]. Balanite A. is one of the widely distributed woody plants in many African countries including Nigeria especially adapting in semi-arid and arid regions of tropical Africa and its subspecies are reported to be found only in India [7,10-12]. Most literature explores the extraction of oil from the seed, use of Balanite pod as a light coarse aggregate in construction [13,14]. The purpose of this research work investigates the possibility of the calcined endocarp of the Balanite *Aegyptiaca* fruit employed as a cement replacement material and the impact on the properties of cement.

The problem of disposal of agricultural wastes has begun to give great concern globally which could be detrimental to human health. Similarly, due to high cost of cement production coupled with released CO₂ emission tends to affect the greenhouse resulting in global warming. Other issues include the high cost of electricity mounting pressure on the use of wood resources stemming from the high energy demand from the growing population. Thus, forcing researchers to seek for alternatives to prevent over harvesting and deforestation [15]. Thus, there is a need to employ the use of biomass is imperative from other sources other than wood such as endocarp of balanite pod. The energy intensiveness of the cement industry coupled with the need to produce cement blends with better technical properties has provided the opportunity to replace endocarp of balanite pod with wood which is readily available and possess a high calorific value which could be employed as fuels and the resultant ash can serve as a cement replacement material.

Researchers have already been expanding innovative technologies to achieve sustainable raw materials in

the construction and building industry. One of the ways through the use of agricultural waste can be utilized and employed as construction materials. Balanite seeds can be considered as a renewable source and possesses the potential to be employed as a cement replacement material in the building industry in Nigeria. It is in this light, that this study investigates the potential of BEPA as cement replacement material in the construction industry. Mangi et al. [16] stated that the concrete industry consumes a huge chunk of natural aggregates and suggested that the potential of using date palm seed for structural lightweight concrete as well as cheap alternative to gravels, but its durability as an aggregate was called in to question due to the inability to withstand exposure to marine or hydraulic structures [17-21] also proved that the utilization of ash-based cementitious materials is not only environment-friendly but possessed more or less same or better properties compared to conventional concretes/mortars. Previous works have studied the use of other pods like date palm pod ash, palm kernel seed ash, locust bean pod ash etc. as cement replacement material with various success but no work has investigated the use of the ash of endocarp of Balanite pod as a cement replacement material as well as the impact on cement properties. Thus, there was a need to also explore the optimum dosage of these local material which is readily available.

The quality of the cement was blended between 0 -12.5 wt.% at interval of 2.5 wt.% were assessed based on various properties such as consistency, setting time, mortar compressive strength, water absorption according to ASTM standards. This research tries to understand the chemical composition of the BEPA and the impact of replacement of cement with the ash on the cement quality and finally determination of the optimal cement replacement for the mortar strength as well as the water absorption rate.

2. Materials and Method

The materials employed for this study are Portland limestone cement was obtained from Dangote Plc which belongs to class CEM II 42.5R A-L while Balanite seed which was sourced at Damaturu market in Yobe State which was calcined to obtain BEPA. Standard sand was obtained from sieving sand into 3 categories comprising between less than 2 mm IS sieve and residue of 90 microns IS sieve. Sieve ranges include less than 2 mm but greater than 1 mm (33.33%), less than 1mm but greater than 600 microns (33.33%) and less than 600 microns but greater than 90 microns (33.33%). The specific gravities of the 3 divisions of the sand were 2.77, 2.67 and 2.59 respectively. Table 1 summarizes the chemical

composition of Portland limestone cement and BEPA using X-ray Spectrometer. The standard Bogue calculation is significantly useful in determination of the proportion of the four minerals in Portland cement:

$$C3S = 4.0710CaO - 7.6024SiO2 - 1.4297Fe2O3 - 6.7187Al2O3 \quad (1)$$

$$C2S = 8.6024SiO2 + 1.0785Fe2O3 + 5.0683Al2O3 - 3.0710CaO \quad (2)$$

$$C3A = 2.6504Al2O3 - 1.6920Fe2O3 \quad (3)$$

$$C4AF = 3.0432Fe2O3 \quad (4)$$

Table 1. Chemical composition of Portland limestone cement and Balanite endocarp pod ash

Compound	PLC	BEPA
SiO ₂	13.39	70.26
Al ₂ O ₃	4.20	1.12
Fe ₂ O ₃	1.95	2.86
CaO	42.14	11.94
P ₂ O ₅	0.18	-
K ₂ O	0.63	3.92
TiO ₂	0.19	0.00
MgO	0.74	0.74
MnO	0.10	-
Na ₂ O	0.09	0.34
SO ₃	1.03	0.38
Cl	0	0.03
LOI	34.67	8.42
Total	47.49	100.00
SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃	35.26	74.24
C ₃ S	38.75	-
C ₂ S	9.16	-
C ₃ A	7.83	-
C ₄ AF	5.93	-

Balanite seeds were collected from Damaturu, Yobe State, were first open- dried under the sun for a longer period. The dried Balanite pods were then crushed to separate the seeds from the pods and the pods (endocarp) was then calcined with a furnace at 600 °C for 3 hours and the resultant ash was ground to powder and sieved with 90-micron sieve. Characterization of the BEPA was conducted using X-ray Fluorescence spectrometer to determine its chemical composition. Table 2 presents the physical properties of the fine aggregate or standard sand employed in mortar preparation.

Table 2. Physical properties of Standard sand

Parameter	Standard sand
Appearance	Brownish solid
Specific gravity	2.67
LOI	0.27

The experimental matrix for the determination of the physical and mechanical properties of Portland limestone cement and BEPA cement blends are presented in Table 3.

Table 3. Experimental matrix for BEPA cement blend physicommechanical properties

S/No	Cement Blends	PLC wt.%	BEPA wt.%
1	PLC	100	0.0
2	2.5BEPA	97.5	2.5
3	5BEPA	95	5.0
4	7.5QBEPa	92.5	7.5
5	10BEPA	90	10.0
6	12.5BEPA	87.5	12.5

Figure 1 depicts the washed Balanite seed that was open dried while Figure 2 shows the calcination of the separated Balanite endocarp pod in a muffle furnace. Figure 3 shows the BEPA obtained from calcination in the muffle furnace at 600 °C for 3 hours.



Figure 1. Washed balanite seed



Figure 2. Calcination of balanite pod



Figure 3. Balanite endocarp pod ash



Figure 4. Portland limestone cement

The Vicat method was specified by the ASTM, C-618^[22], was used to determine the standard consistency of blended cement paste. Three hundred grams (300 g) of cement blends was mixed with water to form paste which were gauged until the resistance to penetration of a plunger reaches between 5 mm - 7 mm from the bottom of the Vicat mould. The ratio of the water required to the weight of cement which was expressed as a percentage was taken as the standard consistency of the cement.

$$\text{Standard Consistency (\%)} = \frac{\text{Weight of water added}}{\text{Weight of cementitious material}} \times 100 \quad (5)$$

The Vicat method was also employed to determine consistence and both setting times specified by ASTM C 187^[23], ASTM C 191^[24] respectively. The water consistence of the various cement blends obtained were then used to determine the initial setting time of the various cement blends and their pastes were gauged into the Vicat mould. The blended cement paste was placed under the Vicat apparatus with the needle provided for the initial setting time determination. The needle was then lowered gently into the contact with the surface of the test block and quickly released and allowed to sink in. This

process was repeated until the needle did not penetrate beyond a point approximately 4 mm from the bottom of the mould. The period elapsing between the time when the water was added to the cement and the time at which the needle ceased to pierce beyond 4 mm from the bottom of the test block was noted as the initial setting time $T_2 - T_1$. The needle for initial setting time was replaced with annular attachment to determine the final setting time. When the needle did not make an impression on the needle was gently applied on the surface of the test block. The final setting time is the time from when water was added to the cement blend and when the needle ceased to pierce the test block $T_3 - T_1$. Where T_1 refers to the time at which water was first added to the cementitious material, T_2 refers to the time when the needle fails to penetrate 5 mm - 7 mm from the bottom of the Vicat mould while T_3 refers to the time when the needle made an impression but the attachment fails to do so.

The water absorption test was conducted on mortars according to BS 1881-122^[25]. Triplicate of mortar cubes after casting were immersed in water for 28 days curing. These specimens were then removed from the curing tank and oven dried for 24 hours at the temperature 85 °C until the mass became constant and again weighed. The weight was noted as the dry weight of the specimen. After that the specimen was kept in water at 85 °C for 24 hours. Then this weight was noted as the dry weight of the specimen.

$$\% \text{ Water Absorption} = \frac{\text{Weight wet} - \text{Dry weight}}{\text{Dry weight}} \times 100 \quad (6)$$

The compressive strength tests were conducted on moulds of 50 mm mortar cube in accordance with ASTM C 109^[26]. A mix ratio of water, cement and standard sand (1:2:4) was adopted to obtain mortars in which the cement was gradually replaced with BEPA between 2.5 wt.% - 12.5 wt.% at interval of 2.5 wt.% at curing age of the specimen at 3, 7, 28 and 60 days respectively. The mortars were homogeneously mixed and placed in moulds and set on a metal slab and bolts on the moulds were fasten with spanner. The cubes were prepared in triplicates at an average temperature of 20 °C and relative humidity of less than 50%. The assembled moulds were then placed in a vibrating machine and securely held in place while filled with the mortars. The moulds were then vibrated with the aid of a jolting machine for 2 minutes, followed by the specimen being covered with an impervious sheet to avoid evaporation and were cured at room temperature for 24 hours. The cubes were then demoulded after 24 hours and were designated by codes for identification. The mortar cubes were then cured in a tank with distilled water at various ages of 3, 7, 28 and 60 days. The compressive strength test for the mortar was obtained using Tonic Technic compression and machine for triplicates of the cubes which were crushed to

obtain an average strength for the various cement blends.

$$\text{Compressive strength} = \frac{\text{Load}}{\text{Cross sectional area}} \quad (7)$$

3. Results and Discussion

From the chemical analysis, Portland limestone cement is an innovative cement that contains between 5 wt.% - 15 wt.% well ground limestone. The percentage of lime and silica were found to be lower compared to the specification ranges for ordinary Portland cement whereas, the other oxides all fall within the range as reported by British standard BS 12 [27]. Based on the X-ray Fluorescence result obtained, it was clearly seen that BEPA significant silica present in its composition and thus can serve as a suitable cement replacement. It was also observed that BEPA obtained from calcination of the endocarp of Balanite seed at 600 °C for 2 hours consisted of more than 70 wt.% of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ (74.24 wt. %) while CaO content was 11.94 wt.% which met class C with CaO content > 10%, hence the ash satisfies as a pozzolan according to ASTM C 618, thus possessing both self-binding and pozzolanic properties [22,31]. The loss on ignition of BEPA was 8.42% indicating a high degree of unburnt carbon of ash [28,29] which is also responsible for increase in the water requirement due to the high porosity of the BEPA particle resulting in mixture segregation which agrees with Freeman et al. [30], Kaya [31], Olubajo and Osha [32], Olubajo et al. [33]. Table 4 indicates the effect of replacing Portland limestone cement with BEPA at replacement intervals of 2.5 wt.% from 2.5 wt.% - 12.5 wt.% on consistence, initial and final setting time and water absorption of mortars.

Table 4. Influence of BEPA on the consistence and setting times and water absorption of cement bends

BEPA content (wt.%)	Consistency (%)	Water demand (mL)	Initial Setting time (min)	Final setting time (mm)	Water Absorption (%)
0.0	30.0	135.0	147	177	7.10
2.5	31.4	141.3	186	202	7.47
5.0	31.2	140.4	170	185	8.71
7.5	31.0	139.5	176	190	9.25
10.0	31.6	142.2	165	186	10.04
12.5	31.6	142.2	158	186	10.06

Figures 5 and 6 indicate the effect of BEPA content on the water absorption and consistence of BEPA - cement blend respectively. It could be observed from Figure 5 that as cement was gradually replaced with BEPA from 0 - 12.5 wt.% at 2.5 wt.% led to an increase in the rate of water absorption. This increase in the water absorption value observed could be attributed to high loss of ignition

owing to the high carbon content present in the BEPA. Portland limestone cement produced the least water absorption of 7.1% as against 10.06% for 12.5 wt.% with BEPA which increased by 42.5% indicating that the ash content significantly affects the water absorption of cement blended with BEPA. According to Olubajo [34], Olubajo & Odey [35], Sprung & Siebel [36] suggested that the particle size distribution of the ash could influence the strength which in turn affects the rate of water absorption of blended cement mortars. An increase in the water absorption at 28 days was experienced as the cement replacement level with BEPA was increased from 2.5 wt.% - 12.5 wt.% which was contrary to Chai et al. [37] findings. This increase in the water absorption could be linked with increase in the water demand, retardation of its setting times and finally lower strengths. The significant particle pores in the mortars could be linked with the high BEPA content culminating in formation of less dense structure thus allowing water to penetrate easily. Chai et al. [37] also suggested that the relationship between the rate of water absorption was inversely proportional to the mortar compressive strength which agrees with this work with an increase in the rate of water absorption and a reduction in the 28 days strength as the cement replacement level was increased.

Similar trend was observed for BEPA-cement blends requiring more water to attain consistence in comparison with control and cement blended with other ashes such as locust bean pod ash [29], sugarcane bagasse ash [33], coal bottom ash [32,38]. The impact of the BEPA on the water requirement increased from 31% - 31.6% (90 mL - 95 mL) as the cement replacement was increased between 0 - 12.5 wt.%. This increase in the water consistence could be attributed to the high loss on ignition of the BEPA arising from carbon content present in the BEPA incorporated into the cement blend. Thus, inclusion of BEPA into the cement matrix resulted in a higher water consistence compared with control.

Table 5 tabulates the mortar strength of cement blends as a function of the cement replaced with BEPA content as well as their percentage strength gain in comparison with control strength while Figures 8 and 9 depict the effect of cement replacement and curing age on the mortar compressive strength of BEPA-cement blends. For 3 days strength, it could be seen that as the cement replacement level was gradually increased, the various cement blends produced a better strength gain compared with control by 7.80% and 9.85% for 2.5 wt.% and 5 wt.% cement replacement respectively. Whereas, beyond 5 wt.% cement replacement produced lower strengths in comparison with control with strength reduction for

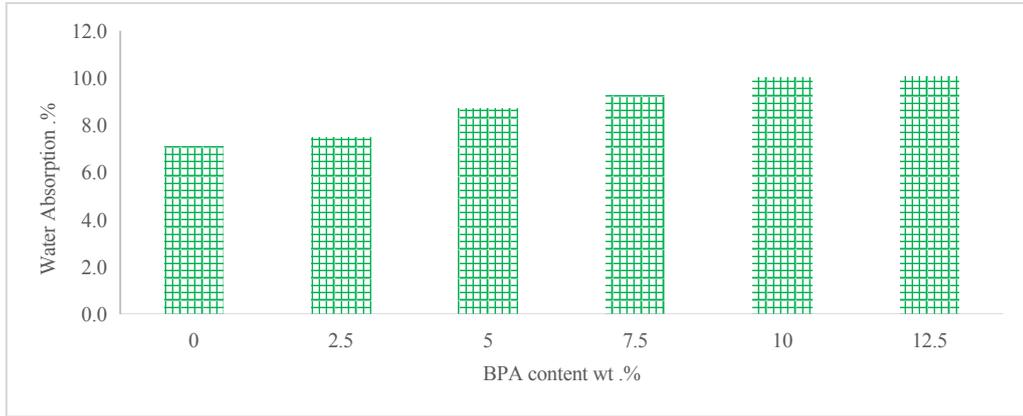


Figure 5. Effect of BEPA content on Water Absorption of cement blends

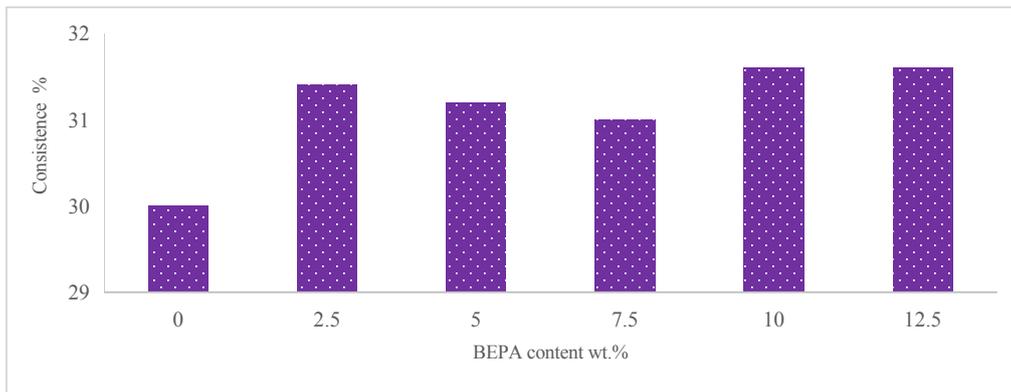


Figure 6. Effect of BEPA content on water consistence of cement blends

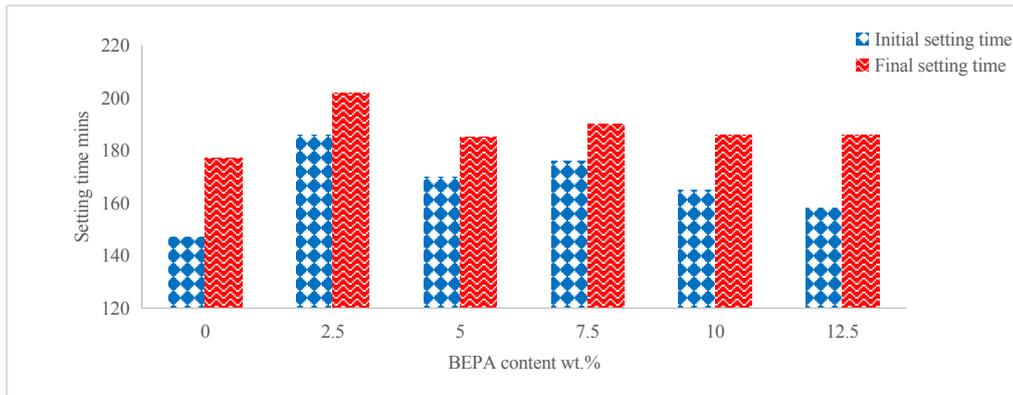


Figure 7. Effect of BEPA content on Setting times of cement blends

7.5 wt.% (15.34%), 10 wt.% (12.99%) and 12.5 wt.% (23.83%) respectively. The increase in the strength could be linked with pozzolanic activity which occurs despite the reduction in the tricalcium silicate/dicalcium silicate (C_3S/C_2S) content, thus resulting in slight increase at lower cement replacement of BEPA. The C_2S , C_3S , are part of the four main minerals were obtained from standard bogue calculation to determine their approximate proportions which gives an idea of the quality of the

clinker/cement. Whereas, at high cement replacement with BEPA produced lower 3 days strength as a result of diminution of clinker effect owing to significant reduction in the C_2S/C_3S content which is responsible for early strength.

The 7 days strengths experienced a decrease in its strength by 39.89% as cement was gradually replaced with BEPA whereas, the 28 days strength of BEPA-cement blends were lower than the control. This reduction

in strength at 7 and 28 days could be attributed to the diminution of the clinker content resulting in formation of limited CSH compared to control as reported by Olubajo and Osha [32]. Another reason for the lower strengths could be due to presence of the unburnt carbon in BEPA, resulting in higher water consistence, retarded setting times culminating in lower compressive strengths as the BEPA content was increased. It could be observed from Figure 8 that the 60 days strength of cement blended with 2.5 wt.% - 7.5 wt.% BEPA produced a better strength gain compared with control of 41.9 N/mm² as against 43.88 N/mm² (4.73%), 43.66 N/mm² (4.20%) and 42.30 N/mm² (0.95%). A reduction in the strength by 9.31% and 20.86% was observed for cement replacements for 10 wt.% and 12.5 wt.% respectively. The initial strength gain experienced between 2.5 wt.% - 7.5 wt.% BEPA could be linked with pozzolanic

activity exhibited by the inclusion of BEPA content resulting in the production of CSH despite diminution of the clinker content which agrees with Olubajo et al. [39].

Figure 9 depicts the mortar compressive strength of various BEPA cement blends at various cement replacements as a function of curing age ranging from 3 days to 60 days. It indicated that the strength of blended mortars exhibited a direct relationship with the curing age as the hydration process progresses for various cement replacement. This is in agreement with Olubajo et al. [39] in which also experienced similar trends suggesting that despite diminution of the clinker content, apart from the hydration reaction taking place, slow pozzolanic reaction occurs between the excess lime and silica presence in the ash to produce more CSH, thus resulting in mortars developing excellent strength in comparison to control.

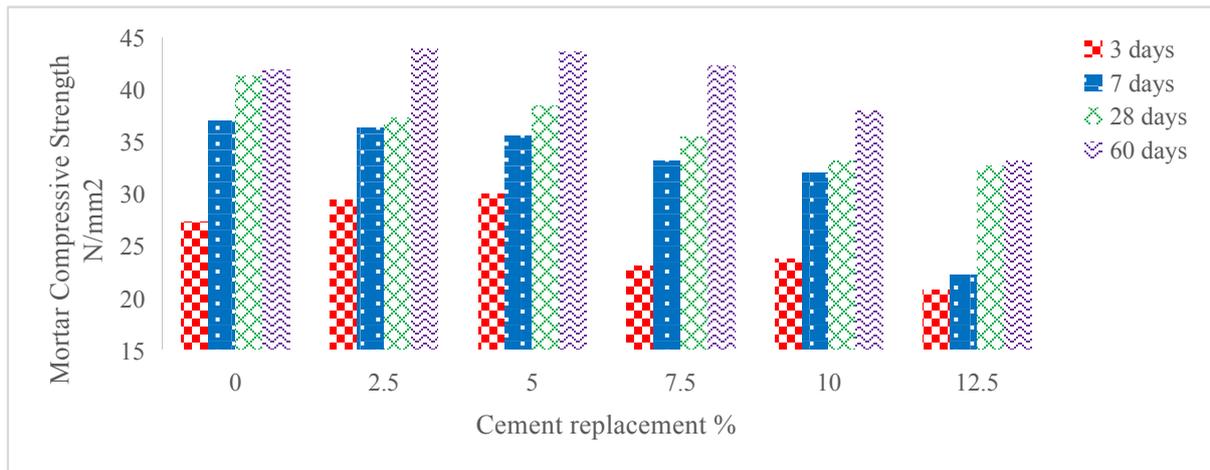


Figure 8. Effect of cement replacement on the mortar compressive strength of various cement blended BEPA and curing days

Table 5. Compressive Strength of mortar for various percentage replacements at different curing days

Cement replacement wt. %	Compressive Strength N/mm ²							
	3 days	Strength gain (%)	7 days	Strength gain (%)	28 days	Strength gain (%)	60 days	Strength gain (%)
0	27.32	100.00	37.05	100.00	41.3	100.00	41.9	100.00
2.5	29.45	107.80	36.32	98.03	37.3	90.31	43.88	104.73
5	30.01	109.85	35.55	95.95	38.49	93.20	43.66	104.20
7.5	23.13	84.66	33.17	89.53	35.52	86.00	42.3	100.95
10	23.77	87.01	32.01	86.40	33.2	80.39	38	90.69
12.5	20.81	76.17	22.27	60.11	32.72	79.23	33.16	79.14

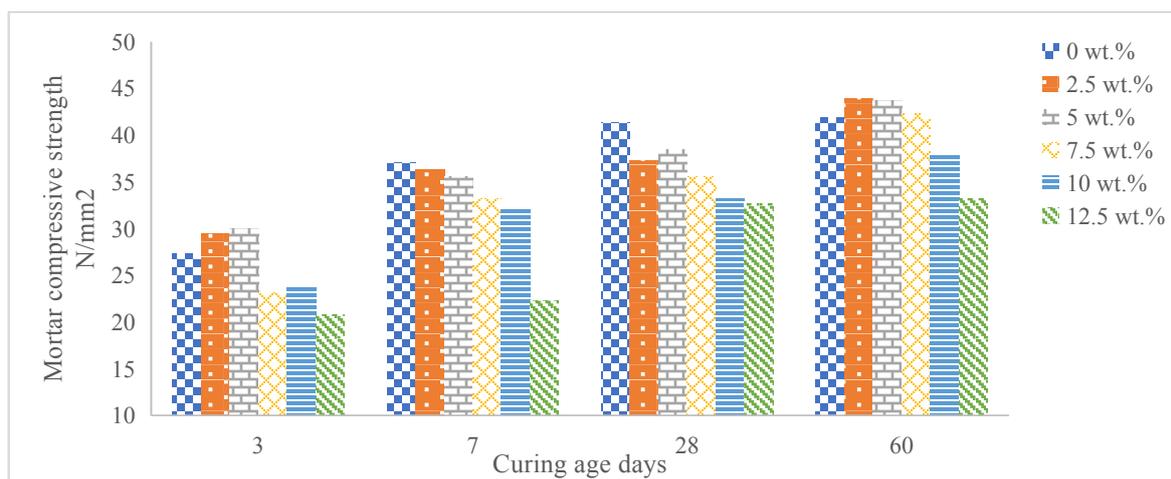


Figure 9. Effect of curing age on the mortar compressive strength of various cement blended BEPA at different cement replacements

4. Conclusions

From this study, it was concluded that BEPA are suitable to be employed as partial cement replacement as a construction material. The chemical composition of BEPA indicated majority of its content comprising of silica, alumina and ferric oxide up to 76 wt.% with CaO content more than 10 wt.%, thus classifying it as Class C pozzolanic material thus having self-binding properties along with pozzolanic properties. Results indicated that the percentage of BEPA should not exceed 7.5 wt.% for mortar compressive strength beyond which results in significant reduction in compressive strengths in comparison with control especially at 60 days. The water absorption of mortars blended with BEPA at 28 days experienced an increase as the replacement of cement with BEPA which was linked with increase in the BEPA content resulting in higher consistence and setting times. All BEPA cement blends experienced an increase in its strength as the curing days progressed. Cements blended up to 12.5 wt.% BEPA are recommended for application in general construction due to the fairly high C_3S content required for early strength.

Conflict of Interest

The authors declared that they have no conflict of interest.

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