Potentials of *Dialium guineense* Endocarp Ash as a Cement Replacement Material

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Abstract. Agricultural wastes such as *Dialium guineense* Endocarp are often generated in volumes that surpass disposal efforts. This concerns communities because improperly handled agricultural waste can lead to environmental challenges. Research on the use of agro-industrial or natural waste as cementitious materials tends to focus on the ashes from orange peel, locust bean pod, palm oil fuel, rice husk and sugarcane bagasse as partial replacements for cement. However, investigations are limited, focusing on Dialium guineense Endocarp ash (DEA) as an alternative cementitious material to reduce CO_2 emissions and agricultural waste. This study explores the potential of DEA as a partial cement replacement material. Partial replacement of Portland limestone cement with DEA 1, 2, 3, 4, 5 and 6 wt.% for physical properties while mortar strength was varied from 0 -5 wt.%. *Dialium guineense* Endocarp pods were collected in Bauchi state- Nigeria, washed, dried, and grinded, followed by determination of thermal stability of the endocarp via Thermogravimetric Analyzer/ Differential Thermal Analyser (TGA/DTA). The resultant ground endocarp was calcined at 600 °C in a furnace for 4 hours, characterised by its chemical composition and functional groups via X-ray fluorescence (XRF) and Fourier Transform infrared (FTIR) spectrometer, respectively. The mortar strengths of 72 cubes for 3 days, 7 days, 28 days and 60 days were produced and determined with a mixing ratio of 1:2:4 (water: cement: sand) according to ASTM standards. The XRF analysis of DEA revealed that the composition of silicon, aluminium and iron oxides was less than 50 wt.% (24.84 wt.%), which did not meet the minimum requirement by standard to be considered a pozzolan with a high CaO content of 25.58 wt.% and possessed significant K_2O content of 36.03 wt.%, an increase in the standard consistency and retardation of both setting times of DEA cement blends was experienced when the cement replacement with DEA was increased. The consistencies and setting time of the DEA-cement blends were higher than control. This prolonged setting times and higher consistency could be linked with the unburnt carbon presence in DEA. As the curing age progressed, the mortar strength experienced increments despite clinker diminution, suggesting pozzolanic activity. Most DEA cement blends produced enhanced strength at 28 days for cement replacement up to 4%, which led to diminished strengths that produced strength slightly lower than control despite clinker diminution. The optimum percentage of cement to be replaced with DEA was determined at four wt.%. DEA possesses properties that are useful as a partial cement replacement material.

Keywords: Agricultural waste; Cementitious Materials; Dialium Guineense Endocarp Ash; Mortar Compressive Strength; Portland Limestone Cement.

INTRODUCTION

Velvet tamarind (*Dialium guineense*) fruits are essential agricultural products in southeastern Nigeria, where the tree is about 30 meters tall. Five species of *Dialium guineense* in West Africa, also identified as Black velvet, fall under the genus of legumes, the Fabaceae family, and the Caesalpinioideae sub-family. The morphological parts of the *Dialium guineense* include wood, bark, gum, seed, leaves, fruits and flowers, which possess numerous benefits ranging from the source of fuel, furniture, pestle production, artefacts, coffee substitute, vegetable, beverages, jam, flavour in snack and decoration respectively [1]. These fruits are commonly sold in local markets and consumed as a snack by various age brackets; according to [2–4], mothers chew the pulp to improve lactation and treat genital infection in Nigeria. According to [5], pulp is a dietary supplement for rural inhabitants during fruit scarcity or dry season. Velvet tamarind pulps are eaten owing to their refreshing properties and pleasant scorching taste. The pulp ripens between January and April, with peak harvest between March and April [6-7].

There is an increasing demand for cement to meet the needs of developers and the construction sector; thus, durable concrete comprised of cement is required. Concrete and mortar (i.e., produced from ordinary Portland cement) tend to react with acid rain, which results in the wearing away of the cementitious material, thereby affecting the durability of a built structure. Various techniques have been explored to curb this negative effect by introducing or using supplementary cementitious materials (SCMs) either from natural or agro-industrial waste such as orange-peel ash [8], Balanite seed pod ash [9], locust bean pod ash [10-11], palm-oil fuel ash [12], rice husk ash and metakaolin [13-15], saw dust ash [16-17] and Sugarcane bagasse ash [18-21] as partial cement replacement. According to [22], using substitute cementitious material as partial cement replacement is one of the most convenient ways of reducing CO_2 emissions, which is applicable in the cement industry geared towards cost reduction. However, other existing agricultural waste with the potential to reduce CO_2 emissions has not been investigated as an alternative cementitious material. This shows that there is a need to explore other waste materials that possess useful properties and, at the same time, protect the environment.

Dialium guineense barks pose environmental threats such as blockage of drainage systems, which could lead to environmental disasters or flooding that can undermine structures kept in place. According to Das [23], a fully matured tree can produce between 20–250 kg of *Dialium guineense* fruit annually. Every year, tons of agricultural waste from *Dialium guineense* Endocarp are generated, which causes environmental issues due to problems with disposal and pollution. Previous studies on cementitious material from agricultural waste have led to considerable enhancements in concrete performance, green environment, construction savings and working conditions [8, 11, 13, 17, 20, 24, 25]. Thus, it is necessary to determine the chemical composition of *Dialium guineense* Endocarp ash (DEA) and investigate the potential of its biomass as a cement replacement material.

In addition, previous literature has shown that cracks usually occur in buildings easily and could collapse structures owing to the high lime content present in OPC [26]. Therefore, improving the silica content invariably improves the mechanical properties of the blended cement. Successful construction products from agro-wastes, notably rice husk, sugarcane bagasse, and orange peel, amongst others [8, 13, 20], prompted the study of DEA as a cement replacement material and its impact on its cement properties. Amongst the agricultural wastes, velvet tamarind / *Dialium guineense* Endocarp (DE) is often ignored in literature and may possess cementitious properties. Thus, the reason for employing DEA as a cement replacement material is to lower the lime content to forestall the diminution of the durability of structures [26].

The main purpose of SCMs mixed with cement is to enhance the properties of the hardened concrete through hydraulic or/and pozzolanic activity. Pozzolans are mainly finely ground from alumino-siliceous or siliceous materials, which react with lime to form more calcium silicate hydrate and other cementitious compounds in the presence of moisture. Authors [27] state that pozzolans can be grouped into SCMs and mineral admixtures and included in cement. SCMs are employed to enhance the cementitious property and their inclusion either by addition or partial cement replacement, which depends on the material properties and the desired impact on the cement. The testing of material to determine the adequate blending ratio is required to establish the optimum dosage since cementitious materials react differently for various types of cement. Researchers explore blending more than one material with available materials to determine the optimal blending combination. According to [28, 29], variation in the quantity of ash included and the ash reactivity can produce a desirable impact on concrete. Most literature focuses on *Dialium guineense* as a healthy human fruit, but there is limited work on DEA for cement replacement.

Based on the above premise, this study aims to evaluate the potential of DEA as a cement replacement material. More specifically, this study entails the determination of the functional groups, calcination temperature, chemical composition, and mineralogical composition of the DEA via Fourier Transform Infra-red spectrometer, Differential Thermal Analyzer, X-ray Fluorescence spectrometer, Thermo-gravimetric analysis and X-ray Diffractometer respectively – the determination of the consistency, setting time and mortar strength of DEA-cement blends. *Dialium guineense* plant possesses numerous potentials and untapped resources ranging from treatment of various health ailments in traditional medicine to providing silica content for cement additives. This study covers using Portland limestone cement with 1, 2, 3, 4 and 5 wt replacement. The impact of DEA on physico-mechanical properties was also studied.

MATERIALS AND METHOD

Dangote Portland limestone cement (CEM II A-L 42.5R) was purchased from a typical Muda Lawal Market Bauchi sale store. In contrast, *Dialium guineense* fruits were purchased from Yelwa Market in Bauchi State. Standard sand was prepared by sieving sand obtained according to Indian standards, which was gathered and oven-dried to remove moisture before being stored to determine compressive strength. Figures 1-3 depict the *Dialium guineense* fruits, *Dialium guineense* powder and *Dialium guineense* endocarp ash, respectively.

Figure 4 illustrates a flowchart describing DEA preparation and determining the DEA cement blend's physical and mechanical properties. The *Dialium guineense* endocarp (DE) was separated from the pulp, and unwanted debris was also present.

Figure 1 – *Dialium guineense* fruits

Figure 2 – *Dialium guineense* powder

Figure 3 – *Dialium guineense* Ash

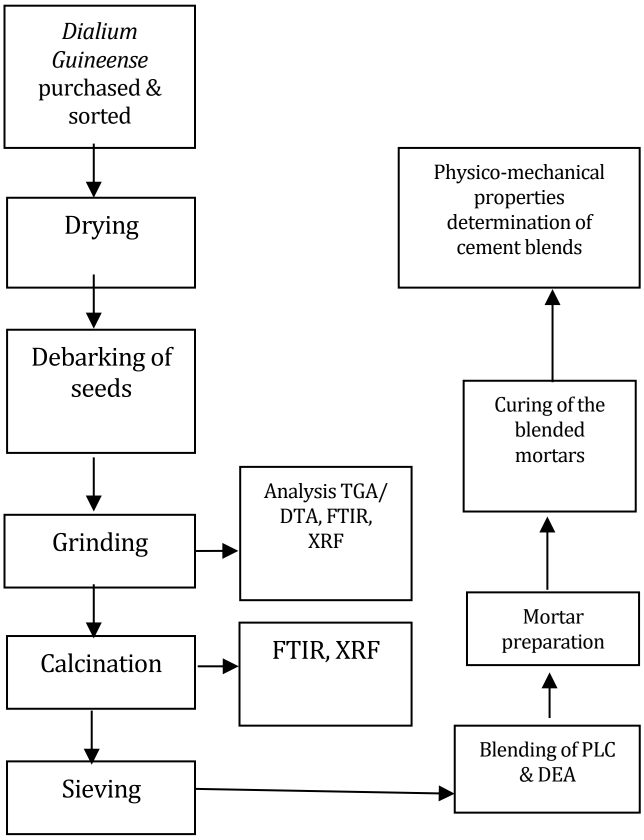

Figure 4 – Flowchart of research methodology for the Preparation of DEA

After this process, the sample was sun-dried to minimise the moisture content, followed by size reduction of the DE with mortar and pestle to fine powder.

Table 1 –Oxides of raw *Dialium guineense* Endocarp (RDE) & *Dialium guineense* Endocarp ash (DEA)

Oxides	RDE, wt.%	DEA, wt.%
SiO_2	34.67	19.47
Al_2O_3	0.18	3.22
Fe_2O_3	1.30	2.15
CaO	2.78	25.58
P_2O_5		6.54
MgO	0.28	-
MnO		1.21
K_2O	2.90	36.03
Na_2O	0.14	-
SO_3	0.50	1.93
Cl	0.013	1.33
SnO_2		1.70
TiO_2		0.53
LOI		0.31

The resultant fine powder was then calcined at 600 °C in the furnace for 4 hours. The calcined DE underwent size reduction and was sieved with a 75 µm sieve before being analysed with an X-ray fluorescence spectrometer. The PLC chemical composition was also determined using an XRF spectrometer.

Normal consistency of the various cement blends was determined via the Vicat apparatus according to ASTM C187 [30]. Four Hundred grams (400 g) of the cement blends were weighed and properly mixed to produce a homogeneous cement paste with weighed quantity of water by the mortar design mix. The time from mixing water with cement and admixture was 3 to 5 minutes. The mixed paste was properly placed into the Vicat apparatus mould and was filled to level with a trowel. The Vicat apparatus plunger was set to touch the cement blend paste surface on the Vicat mould. The plunger was then allowed to fall slowly into the blended paste. This above procedure was repeated using other cement blends presented in Table 2 for various water measures until the gauge read between 5 to 7 mm and the water measure was recorded.

Table 2 – Experimental design for Portland limestone blended with DEA at various replacements

No	Cement blends	Cement, wt. g	DEA, wt. g
1	100PLC	100.0	0.0
2	1DEA-99PLC	99.0	1.0
3	2DEA-98PLC	98.0	2.0
4	3DEA-97PLC	97.0	3.0
5	4DEA-96PLC	96.0	4.0
6	5DEA-95PLC	95.0	5.0

The setting time was carried out according to [46]; the normal consistency was determined by recording the time when cement was mixed with water homogeneously. The resultant paste was then placed into the mould via a hand trowel. The paste was placed in the mould, and its surface was levelled, smoothened and then placed under the 1 mm needle. The needle was gently lowered on the blended paste surface and allowed to drop to the bottom of the mould. This procedure was repeated at intervals of 10 minutes till the needle did not exceed 5 mm. The setting time was recorded as the interval between when cement was mixed with water and sufficient stiffness in which the needle did not exceed 5 mm above the bottom of the mould. The final setting time was obtained when the metal annular attachment needle was allowed to touch the surface of the cement paste and make an impression on the cement blend surface without the annular cutting edges visible. The blended cement mortars were prepared according to [31] by mixing DEA, PLC, standard sand, and distilled water. Table 2 presents the experimental mix design comprising the proportions of cement calcined samples.

The chemical composition of raw *Dialium guineense* (RDE) and DEA was determined using the X-ray fluorescence spectrometer. In contrast, the functional groups were determined via Fourier Transform Infra-Red spectrometer for RDE and DEA, respectively. TGA/DTA of the ground RDE sample was determined using the Thermogravimetric and Differential Thermal analysers, respectively. The significance of these analyses was to investigate RDE and its volatile fractions' thermal stability through differential changes in the weight of a given sample calcined at a constant rate. The TGA spectra of RDE were determined by heating from 30-900 °C at 10 °C/min at Ahmadu Bello University Zaira, Kaduna State. TGA and DTA methods are most popularly employed to determine the thermal stability of RDE. TGA is a procedure that studies the changes in the mass of a sample as a function of time and temperature and enables the valuation of the weight loss of materials. DTA provides temperature differences between the reference material and sample measured [32].

RESULTS AND DISCUSSION

The thermogravimetric curve represented by the red curve obtained for RDE in Figure 5 indicates three substantial mass loss stages: firstly, at 150–380 °C signifies by drying (capillary pure residual water), an endothermic peak and ettringite dehydration. This first mass loss stage is characterised by several minor stages, which mostly occur, including capillary pore water, interlayer water and adsorbed water. Secondly, the mass loss stage or thermal stability point is around 500–600 °C, which is the dehydration temperature. Thirdly, the mass loss stage or thermal stability point at 700–887.4 °C can be attributed to the decarbonisation temperature [33]. Figure 5 shows the spectra generated for TGA/DTA by heating from 30–900 °C at 10 °C/min. The DTA curve, represented by the blue curve, revealed that the three mass loss stages correspond to an endothermic process because the system absorbs energy from the surroundings in heat. The DTA curve can be identified by a U-shaped depression in the curve, which symbolises rapid mass loss followed by rapid mass gain. The sample RDE was burnt at 600 °C for four hours from the above result. This was carried out because 600 °C was within the range of the thermal stability of the sample. This is similar to previous literature on biogenic material burnt at 600 °C [34].

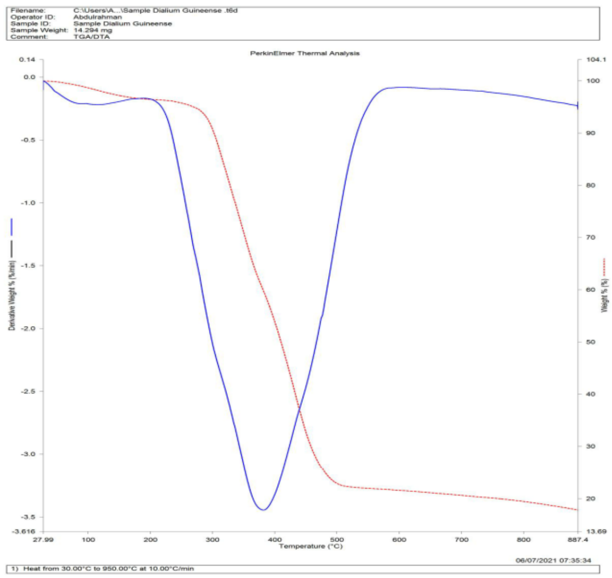

Figure 5 – Thermogravimetric / Differential Thermal Analysis for RDE

The chemical composition for the RDE and DEA, calcined at 600 °C for four hours, was obtained via X-ray fluorescence spectrometer at BUA Cement Company Plc at Edo state. Table 1 indicates the chemical compositions of RDE and DEA obtained using X-ray Fluorescence Spectrometers respectively, and revealed that the summation of oxides of silicon, aluminium and iron of DEA was 24.84 wt.%, which was less than 50 wt.%, which did not satisfy the requirement to be identified as a pozzolan. CaO content was 25.58 wt.%, which fell within Class C pozzolan between 10-30 wt.% CaO. Other oxides include SO3 content, which was 1.98 wt.% (less than 5%), whereas the LOI was 0.31 wt.%, in which most requirements met standards according to ASTM C 618 [35]. Similar lower oxide content summation was observed according to the works [11, 36] for Locust bean pod ash (LBPA).

The FTIR results indicate five band ranges: the band 3500-4000 cm^{-1} specifies the loss of $Ca(OH)_2$; 1600-3500 cm^{-1} indicates the widening of the (OH-) bond and bonding of the (H-O-H) vibrations, 1000-1600 cm^{-1} indicates the gain of the (Si-O-Si) bond typical of quartz, 1000 cm^{-1} indicates the symmetric widening of the (Si-O-Si) and

(Al-O-Si) bonds and the fifth bond below 500 cm^{-1} indicate bonding of the (Si-O-Si) and (O-Si-O) bonds [37]. Twelve peaks were observed from the FTIR spectra in Figure 6.

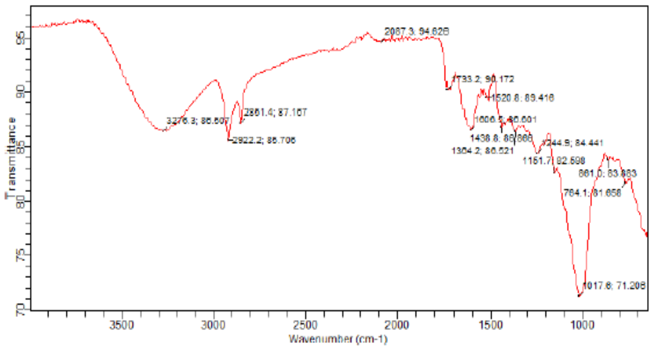

Figure 6 – FTIR spectra for raw *Dialium guineense*

Figure 7 shows the FTIR curve for the DEA at 600 °C for four hours, and it was observed that DEA spectra can be specified by four noticeable peaks at 670.76, 870.76, 982.20 & 1397.80 cm^{-1}, respectively.

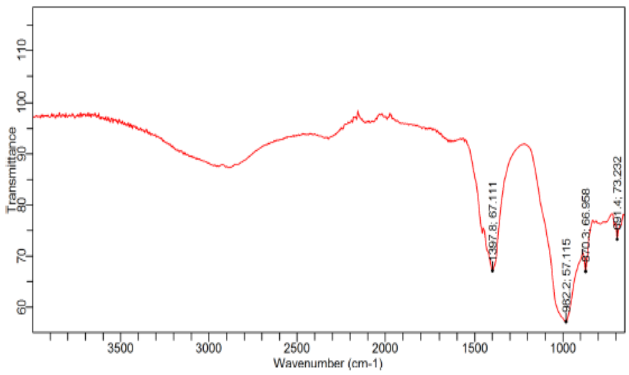

Figure 7 – FTIR spectra for DEA

The FTIR spectra at 870.76 cm-1 fit C-O- v4 CO3, representing CaCO3 with the findings [38]. Bands at 982.20 cm^{-1} also indicated O-Si-O vibration presence attributed to asymmetrical widening bands vibration, which agrees with [39] at 983 cm^{-1}. The wave number band between 1000–1644 cm^{-1} designates the small amount of carbon present for 1397.80 cm^{-1} while O-H-O extending and stretching vibration between 3460 cm^{-1} suggesting free water present similar to works [38, 40], which recorded 3270.76 cm^{-1}. Free water band was observed at about 2922 cm^{-1} due to the widening vibration of H_2O molecules, which also agreed with [41, 42] at 3427–3636 cm^{-1}, which in the range of very broad hydroxyl groups between 2500–3500 cm^{-1}.

The effect of replacing cement with DEA between 0-6 wt.% cement replacement on the normal consistency for various DEA cement blends was determined, and the results were tabulated in Table 3.

Table 3 – Variation of the DEA content on the blended cement consistency and setting time

DEA wt.%	Water, ml	% Consistency	Setting time (initial) mins	Setting time (final) mins
0	155	31.0	172	378
1	175	35.0	190	394
2	180	36.0	195	398
3	185	37.0	200	404
4	190	38.0	176	355
5	190	38.0	180	360
6	195	39.0	188	366

The significance of the consistency test comes into play to determine the amount of water used to make standard paste; this property influences the cement's strength and other properties [43].

Figure 8 indicated that the standard for water consistency experienced increments by 112.9%, 116.1%, 119.4%, 122.6%, 122.6% and 125.8% compared with control as the DEA content was increased from 0–6 wt.%. This increase in consistency could be linked with the unburnt carbon present in DEA, which agrees with [44, 45, 9]. Thus, the water for normal consistency of cement blended with DEA increased as the DEA content was increased.

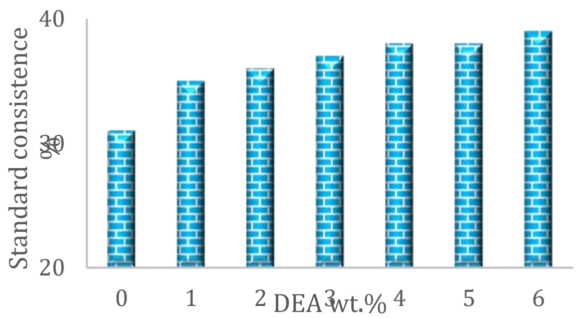

Figure 8 – Standard consistency of cement blends as a function of the DEA content

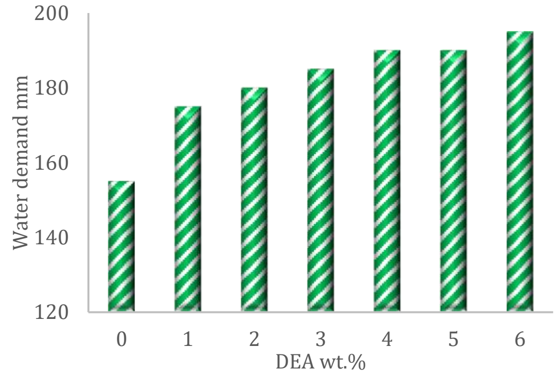

Figure 9 – Water demand of cement blends as a function of the DEA content

Figure 10 illustrates the setting times as a function of the amount of cement replaced with DEA.

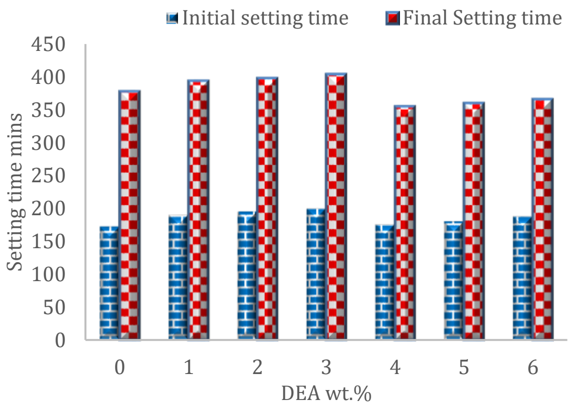

Figure 10 – Setting time of cement blends as a function of the DEA content

All cement blended with DEA produced higher setting time results as the DEA content increased compared to the control, 2.33% and 16.28% (3 wt.% DEA). The increased water demand and retardation of the setting times observed might be related to the presence of unburnt carbon in DEA along with clinker reduction. Whereas cement was gradually replaced with DEA, it led to an increase in the final setting time up to 3 wt.%, beyond which any further increment led to a decrease in its setting time compared to control. The reduction in the setting time of the DEA cement blend could be attributed to the available lime present in DEA, resulting in an enhanced hydration rate despite the diminution of clinker content as the DEA content was increased, which agrees with [47].

Cement replacement of PLC with DEA for 0, 1, 2, 3, 4 and 5 wt.% were prepared as mortars at various curing ages at 3 days, 7 days, 28 days and 60 days, and their compressive strengths were determined respectively and tabulated in Table 4. Most cement blends exhibited excellent percentage strength gain compared to 7 days and 28 days control strength, whereas mortar compressive strength for 3 days produced between approximately 77.03% and 107.62%, respectively. 7- and 28-day strengths produced a higher strength index than control, indicating that the pozzolanic activities are evident for 7 days and 28 days strength despite clinker diminution. Most DEA cement blends produced excellent strength indexes for 7 and 28 days, as shown in Table 4, evident by indexes above 100%.

Table 4 – Compressive strength of DEA-Cement Blend

Sample	Wt.%	3 d Strength, N/mm^2	% Strength Gain	7 d Strength, N/mm^2	% Strength Gain	28 d Strength, N/mm^2	% Strength Gain
1	0	19.68	100.00	20.00	100.0	28.04	100.00
2	1	21.18	107.62	31.88	159.4	32.76	116.83
3	2	19.70	100.10	26.60	133.0	33.82	120.61
4	3	15.16	77.03	25.80	129.0	39.85	142.12
5	4	15.20	77.24	25.08	125.4	28.16	100.43
6	5	18.46	93.80	23.60	118.0	26.28	93.72

Data showed that as the curing age progressed, it resulted in an increment in mortar strength at various cement replacements, which agrees with various ashes such as coal bottom ash [24], Locust bean pod ash [11], rice husk ash [13], fly ash [44–45]. This trend can be attributed to silica reacting with lime as the curing age lengthened. The pozzolanic effect was minimal because the DEA was around 58.72 wt.%. The results indicated that as cement was replaced with control, 1%, 2%, 3%, 4% and 5% DEA strengths improved by 42.48%, 54.67%, 71.68%, 162.86%, 85.26% and 42.36% with lengthening of curing period from 3 to 28 days respectively. The optimal DEA content required to obtain strength slightly above the control was 4%, which produced 93.72% of the control.

CONCLUSIONS

Based on the findings of this study:

1. The TGA/DTA was employed to determine the calcination temperature of RDE at 600 °C. The chemical composition of DEA via XRF revealed that the summation of silicon, aluminium and iron oxides was less than 50 wt.% (24.84 wt.%), which did not meet the minimum requirement according to ASTM C to be considered a pozzolan with a high CaO content of 25.58 wt.% and possessed significant K2O content of 36.03 wt.%.

2. Results revealed an increment in the standard consistency of DEA-cement blends as the cement replacement with DEA was increased from 0–6 wt.%. All cement blended with DEA possessed higher standard consistency and elongated setting times, which could be accredited to unburnt carbon content, dependent on the quantity of cement blended with DEA.

3. As the curing age was lengthened, increments in most of the mortar compressive strength of DEA-cement blends were observed irrespective of the increase in the cement replacement level, thus indicating evidence of pozzolanic activity. The mortar strength of 7 days for all the cement blends exhibited excellent strength compared to 7 days control strength, thus affirming pozzolanic activity despite the diminution of clinker content. The mortar compressive for 28 days experienced enhanced strength compared with the control up to 4 wt.%, which produced slightly lower strength than the control.

4. Studies have indicated that DEA can be a partial cement replacement material. The optimal DEA content replaced by cement was obtained at 4 wt.% replacement.

Conflict of Interest

The authors declared that they have no conflict of interest.

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