Mechanical and structural characteristics of cement mortars blended with locust bean pod ash

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Abstract: The need to reduce the environmental pollution resulting from agro-wastes and to source a material that can be used to replace cement in order to reduce the pressure on its consumption necessitated this research. This study looks at the effect of cement replacement with locust bean pod ash (LBPA) as a supplementary cementitious material on the mechanical and structural characteristics of mortars. The fresh properties (workability, initial and final setting times), compressive strength at 7, 14, 21 and 28 days and microstructural analysis by scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), and X ray diffraction analysis (XRD) were evaluated. LBPA were considered at replacement levels of 0, 10%, 15%, 20% and 30% of cement mass for preparation of the mortar samples. Workability of the cement mortars reduced as the content of LBPA increases while initial and final setting times increased in relation to increase in LBPA content in the matrix. An increase of about 79% in compressive strength at 7 days, 100% at 14 days, 147% at 21 days and 136% at 28 days were recorded with LBPA content of 15% LBPA being the optimum level when compared to control mix. Maximum compressive strength ranged between 38.3 and 65 MPa after 7 to 28 days curing. Microstructural analysis revealed less voids and pores, and the presence of dense CSH gels which helped to maintain the optimum compressive strength at 15% LBPA cement replacement level of the mortar. The results indicated that LBPA is a reactive pozzolanic material and can be used as a supplementary cementitious material for producing medium-strength concrete.

Keywords: compressive, strength, agro-waste, pod, ash

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1 Introduction

A pozzolan is described as a siliceous or alumino-siliceous (aluminous and siliceous) material which in itself, possesses little or no cementitious value but which will, in finely divided form and in the presence of water, react chemically with alkali and alkaline earth hydroxide at ordinary temperatures to form or assist in forming compounds possessing cementitious properties (Omoniyi and Akinyemi, 2012). Pozzolanic materials will form calcium silicate cement when they react with soil particles in the water. The cementing agents are the same as in the case of Portland cement, however, in Portland cement, the calcium silicate gel is formed from the hydration of anhydrous calcium silicate (cement).

Due to increasing industrial and agricultural activities, tonnes of waste materials are deposited into the environment with little effective method of waste management or recycling. Some of these deposits are not easily decomposed and their accumulation is a threat to the environment and people at large. Some of these waste

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materials are locust bean pods, bagasse, rice husks, sugarcane straw, bamboo leaf, maize cobs, snail shells, palm-kennel shell, coconut shell, saw dust and groundnut shell (Omoniyi and Akinyemi, 2012; Adama and Jimoh, 2012).

Super plasticizers are chemical admixtures used where well-dispersed particle suspension is required. These polymers are used as dispersants to avoid particle segregation and improve the flow characteristics of suspensions in concrete applications. Their addition to concrete or mortar allows the reduction of the water-cement ratio, not affecting the workability of the mixture, and enables the production of self-consolidating and high-performance concrete.

Increasing population in most developing nations has led to an astronomical increase in demand for cement. Therefore in order to meet this demand, cement productions have been jerked up with its accompanied carbon emissions (Omoniyi and Akinyemi, 2012; Adama and Jimoh, 2012). Supplementary cementitious materials (SCM) have been proffered as the solution to this problem through its effective and successful use in construction materials. The adoption of SCMs in construction leads to a major reduction in carbon emissions into the environment. They are also known as pozollans and if used in combination with cement, they form cementitious particles, but alone without combination with cement, they do not have cementitious elements. SCMs' such as rice husk ash, fly ash, blast furnace slag as well as silica fume amongst others have been utilised (Mahmud et al., 2016; Akpenpuun et al., 2017). Nonetheless, the accessibility of these substances are limited to specific locations and industrialization, therefore it is imperative that focus must be shifted to conducting studies on SCM's emanating from agriculture more than others because of its abundance and availability in most parts of the world (ASTM Standard E986-04, 2017). Other agricultural waste materials used as SCMs include wood waste, bamboo leaf ash and corncob ash. Very limited attention is paid to locust bean pod as another prospective pozollan in cement for building applications being an agricultural waste material. African locust bean (*Parkia biglobosa*) is a perennial deciduous tree of the Fabaceae family. It is found in a wide range of environments in Africa and is primarily grown for its pods that contain both a sweet pulp and valuable seeds. The annual production of African locust bean is about 201,000 tonnes of the bean fruit annually in Nigeria (Sina and Traoré, 2002). Various parts of the locust bean tree are used for medicinal purposes and the crushing and fermenting processes of these seeds constitutes an important economic activity.

The pod is the most economic part of the tree and it is usually opened up in order to remove the pulp and the seeds. Thereafter it is discarded, burnt and the ashes dumped in landfills (Tangchirapat *et al.*, 2009; Adama and Jimoh, 2012). Limited studies had been conducted on its use as supplementary cementitious materials in concrete and the focus of these studies was not on the microstructural analysis such as scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), and X ray diffraction analysis (XRD) and non-use of superplasticizers which improves workability of the mortars produced (Van *et al.*, 2013). In this present experiment, the performance of locust bean pod ash blended cement on workability; setting time and compressive strength of mortars were evaluated while incorporating superplasticizer. Similarly, the use of SEM, EDS, and XRD were employed in further analysing the internal networks and the crystalline materials located within the samples.

2 Materials and methods

2.1 Locust bean pod ash (LBPA)

The waste was obtained from a farm around the University environment. Sorting was done by removing unwanted organic materials such as leaves, shrubs and stalks. LBPA was sun dried for two weeks until the change in moisture content was 0%. 10 kg of it were then calcined in a muffle furnace at 600^oC with heating rate of $40^oC\ min^{-1}$ for 2 hours and 2 hours retention time. The obtained ashes were ground manually for 4 minutes and the particle size analysis was done as shown in Figure 1. Shown in Figures 1 and 2 are the pods and ash. Based on the report of Cordeiro et al. (2012), the ash could be classified as class C (Table 1).

2.2 Fine sand aggregate

The sand used for the study was sourced locally and it conforms to ASTM Standard C778-17 (2017). Sieving was done and the gradation of both fine sand and LBPA has seen in Figure 3. The specific gravity is 2.12, bulk density is 1541 kg m^{-3} and fineness modulus is 2.17.

Figure 1 Locust bean pods

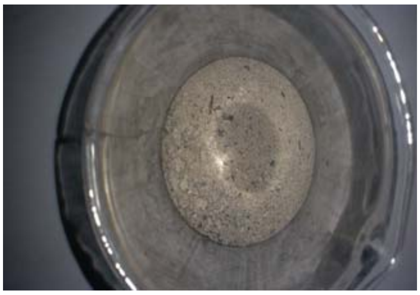


Figure 2 Locust Beans Pod Ash (LBPA) in beaker

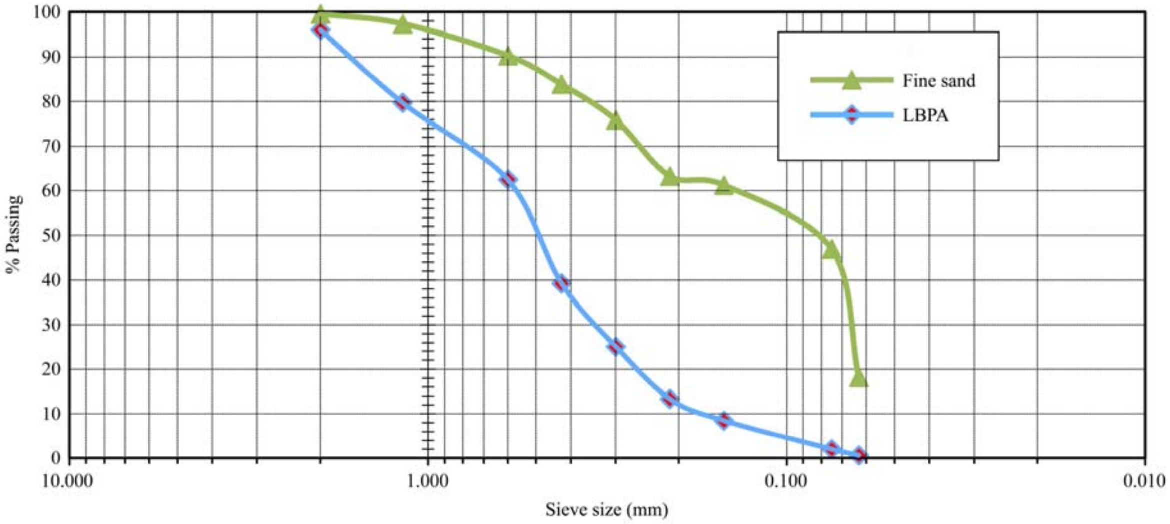


Figure 3 Sieve analysis of LBPA and sand

2.3 Cement

Ordinary Portland cement that conforms to type 1 (ASTM Standard C150/C150M-18, 2018) was used and mixed with water, sand, LBPA and superplasticizer. The chemical composition of both cement and LBPA are shown in Table 1.

Table 1 Chemical composition of cement and LBPA

Oxides	LBPA	Cement
SiO_2	40.25	20.70
Al_2O_3	13.15	5.75
Fe2O3	9.00	2.50
CaO	12.50	64.00
MgO	2.03	1.00
SO_3	1.50	2.75
Loss of ignition	6.05	2.30
Blaine fineness (cm^2 g^{-1})	5502.62	3100.43

Note: Cordeiro *et al.* (2012).

2.4 Admixture

The super-plasticizer (SP) used was Hydroplast – 300, a third generation high performing polycarboxylate ether super-plasticizer with great water reducing properties which allows for the production of high consistency mixed concrete with low water-cement ratio. It also ensures extended workability and improves early and final strength development.

2.5 Mixture proportioning

Five different mortar mixes (Figure 4) were designed for the experiment as shown in Table 2. Replacement levels of 0%, 10%, 15%, 20% and 30% of cement mass were used. The water: cement binder ratio was maintained at 0.55 and a constant dosage of 1% of cement of SP was utilised in order to enhance the workability of the mortar. Mortar samples were prepared for SEM, EDS, as well as for XRD analysis.

Figure 4 Mortar sample

Table 2 Mix design of mortar (kg m^{-3})

Mixture code	Cement	LBPA	Sand	Water	SP (grams)
C_T	628.3	-	1256.4	341.5	-
A	565.5	62.8	1132.3	314.2	5.7
B	534.1	94.2	1069.7	297.3	5.3
C	502.6	125.7	1006.1	279.2	5.0
D	439.8	188.5	880.2	244.3	4.4

Note: A – 5%, B - 10%, C – 15%, D – 30%.

2.6 Preparation and casting of samples

Dry mixing was first performed for both cement and sand manually for 3 minutes, thereafter the LBPA was mixed with it until a homogeneity appearance was attained. The required quantity of super-plasticizer was mixed with a tiny fraction of the water required and added to the mix. The mix was turned and the remaining quantity of water required was added gradually and turned over with hand-trowel until the mix appeared uniform in colour and consistency. A total of twelve 50 mm cube specimens were batched, demoulded and cured for 7, 14, 21 and 28 days in a curing tank containing water at 23°C respectively.

2.7 Testing programme

The adopted testing method used are setting time (ASTM Standard C807-13, 2013), workability (ASTM Standard C1437-15, 2015) and the compressive strength (ASTM Standard C109M-16, 2016) test was performed after 7, 14, 21 and 28 days of water curing using the universal testing machine (UTM) at a loading speed of 10 mm min^{-1} until failure occurred.

2.9 Characterisation of blended cement mortars

Samples were examined using a field emission scanning electron microscope (FESEM) (model: JSM-7600F, Jeol, Japan) at an acceleration potential of 15 kV. The fractured surfaces of the specimens were sputter-coated with a thin layer of platinum using a JFC-1600 auto fine coater. The EDS results were obtained using the same model of SEM machine.

2.8 X-ray diffraction analysis for mineral identification

Powdered samples were pelletized and sieved to 0.074 mm. These were later taken in an aluminium alloy grid (35 mm × 50 mm) on a flat glass plate, covered with a paper and the samples were compacted by gently pressing them with the hand. Each sample was run through the Rigaku D/Max-lllC X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan and set to produce diffractions at scanning rate of 20 min^{-1} in the 2 to 500 at room temperature with a CuKa radiation set at 40 kV and 20 mA.

3 Results and discussions

3.1 Workability

The results of the fresh concrete properties of the LBPA mortars in Figure 5 indicated satisfactory flow of slump in the range between 14 mm and 49 mm. The least workability was gotten at the highest percentage of 30% LBPA while the highest slump flow was obtained at the control mix, 0% cement replacement. This could be interpreted that workability reduces as the content of LBPA increases in the mortar mix. This reduction in flow was similarly reported by Tangchirapat et al. (2009), and Cordeiro et al. (2012), where it was stated that an increase in ash content led to a progressive decrease in workability. The slump values of samples E, D, B, and A decreased 250%, 69%, 44% and 23% respectively as compared to control slump value. This reduction in slump on increase of LBPA could be attributed to the porous nature it has due to the presence of macro and meso-pores located within and on the surface of the material which led to its huge specific surface area. The LBPA will thereafter absorb some amount of water unto its surface during mixing which would cause subsequent reduction in free water and a lower slump value (Cordeiro et al., 2012).

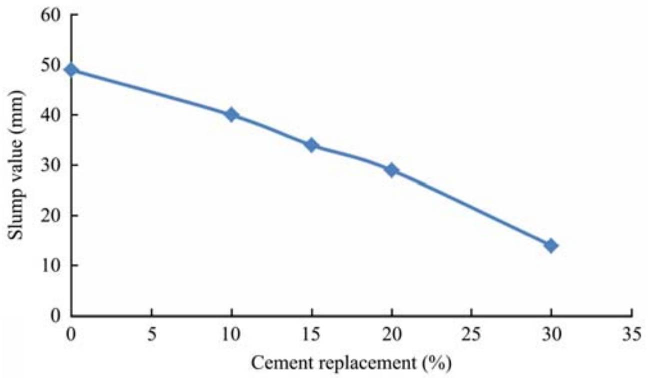


Figure 5 Slump values of fresh concrete

3.2 Initial and final setting times

Figure 6 shows the final and initial setting times. The mean setting time was 174.5±41.7, 204±33.9, 220±28.3 and 263.5±30.4 mins for 0, 10%, 15%, 20% and 30% cement replacement with LBPA. An observed increase in both initial and final setting times as the cement binder replacement increases was noted. At 30% replacement, the LBPA blended cement mortar increased the initial setting time in relation to the control sample by 66.8% while the same replacement level increased the final setting time relative to the control by 39.7%. This is dependent on the temperature during the ashing process, low temperature would yield crystalline powders such as from open burning while high temperature would produce amorphous powders (Tangchirapat et al., 2009). The same trend was confirmed by other researchers that used agro-waste ash in concrete. Another reason adduced for this pattern is that the low rate of cement hydration in the paste consisting of the LBPA pozollan led to an increase in both initial and final setting times on subsequent increase in cement replacement levels (Cordeiro et al., 2012).

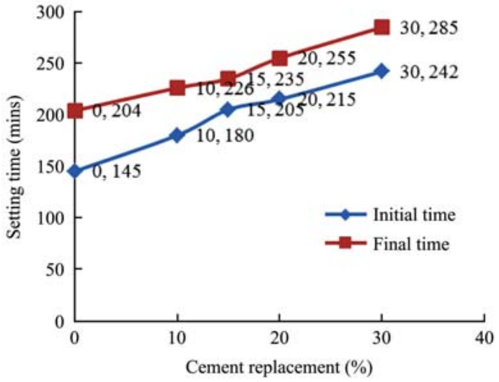


Figure 6 Initial and final setting times

3.3 Compressive strength

The compressive strength in relation to the LBPA cement replacement levels and specimen age (7, 14, 21 and 28 days) is shown in Figure 7 and the samples on which the test was carried out are shown in Figures 3. The mean compressive strength was 22.5±4.86, 14.27±1.99, 13±4.96, 14.8±4.09 and 10.8±0.63 MPa for 0, 10%, 15%, 20% and 30% cement replacement with LBPA respectively. As seen in Figure 4, there was an increase in compressive strength up till the 15% replacement level then a reduction in the values was noticed. At 7 days the compressive strength ranged 10-25 MPa with 0% cement replacement having the highest compressive strength of 25 MPa. The compressive decreased as the cement replacement levels increased from 0 to 30% after 7, 14 and 21 days of curing. However, after 28 days of curing the compressive strength decreased from 15 MPa to 12 MPa (10% LBPA), 9 MPa (20% LBPA) and 11 MPa (30% LBPA) and then increased to 21 MPa (15% LBPA). The results are comparable to Chao-Lung et al. (2011) and Ndububa and Uloko (2015) who reported that the compressive strength decreased with increasing percentage of cement replacement. This improvement is related to reduced pores and increased C-S-H compound in the matrix which resulted into the formation of a dense mortars after the early curing days (Raman et al., 2011). The data from this experiment was compared with similar studies conducted using agro-waste based ash as cement replacement to relate the effects of cement replacement and the age to the compressive strength as shown in Table 3.

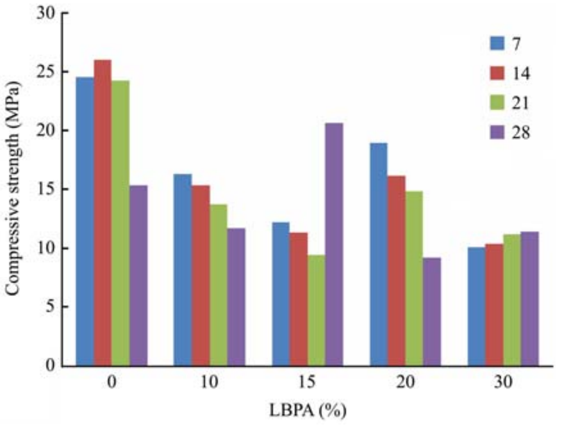


Figure 7 Compressive strength of LBPA blended mortars

Table 3 Comparison with compressive strength of other agro-waste ashes studied

Sources	Agro waste ash	Optimal %	Superplasticizer	Age	Comp. Stre. (MPa)
P.S.	LBPA	15	Yes	28	65
41	Bagasse	10	No	90	68
42	Palm oil fuel	10	No	28	6
43	Rice husk	15	Yes	28	51
44	Banana leaf	7.5	Yes	91	42
45	Bamboo leaf	20	No	28	56

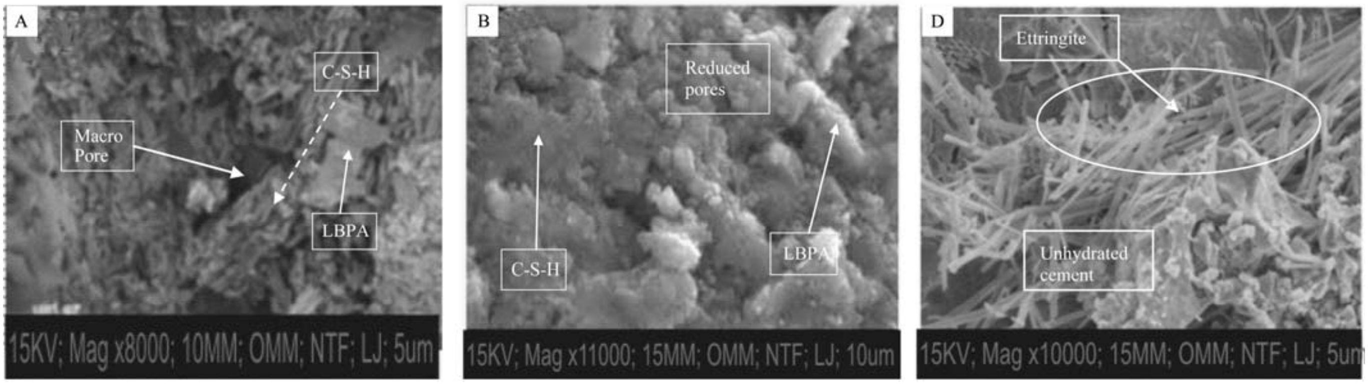
Note: P.S. – present study.

Almost any research conducted on the use of agro-waste material in green concrete included compressive strength or strength activity index as one of the properties evaluated. The reason is that it is one of the major properties used in the design of mortars and concrete for various load applications and as well as a form of field quality control assessment. From the result of previous research shown in Table 3, the compressive strength ranged between 42-68 MPa when other agro wastes were used as compared to 65 MPa using LBPA. In the same vein, the optimal cement replacement across these studies varied from 7.5%-20% which is in tandem with the optimal cement replacement of 15% recorded in this work as well. It has been noted by some researchers that mortars incorporated with SCM tend to develop later strength which would be higher than the control samples without SCM replacement (Raman et al., 2011; Chopra and Siddique, 2015).

3.4 Characterisation of LBPA blended cement mortars

3.4.1 Scanning electron microscope (SEM)

The micrograph of the surfaces of 10% (A), 15% (B) and 20% (D) LBPA mortar samples after 28 days of curing is shown in Figure 8. Figure 8(a) showed presence of both micro and macro pores of sample with 10% LBPA content and C-S-H gel could also be sighted in it. These pores are largely caused by evaporation of water during hydration based on the LBPA content in the sample. It has been stated that agro based ashes used as cement replacement tend to absorb free water into its pores during mixing and this is released at the later stages when the relative humidity of the cement drops. This should cater for the drop in water requested for hydration to be completed but where this free water is not available in required quantity, porous surface may emerge as might be this case under consideration (Chopra and Siddique, 2015; Mahmud et al., 2016). The presence of these voids largely led to the poor compressive strength performance of the sample. However, in Figure 8(b), mix with 15% LBPA replacement could be seen, reduced voids on the surface due to hydration improvement, and likewise large occurrence of C-S-H gels could be noticed as well on the surface. This could be the reason for the optimal compressive strength gotten at this mix level. The increase in LBPA content could possibly improve the pore characteristics because it led to higher amount of C-S-H gel formation during the pozzolanic reaction which contributed to the refinement of pore structure subsequently leading to volume reduction of large pores and porosity of the mortar. Figure 5(c), is the micrograph of 20% LBPA replacement, it was noted the mix commenced disintegration at the quantity of LBPA was increased; this perhaps led to the reduction in compressive strength. Ettringites are the needle like crystals that are developed in vacant spaces during hydration also led to weakening of the concrete strength as well. Some portions of incompletely hydrated cement particles were also identified, this also contributed to lower strength of the mortars.



(a) 10% LBPA (b) 15% LBPA (c) 20% LBPA

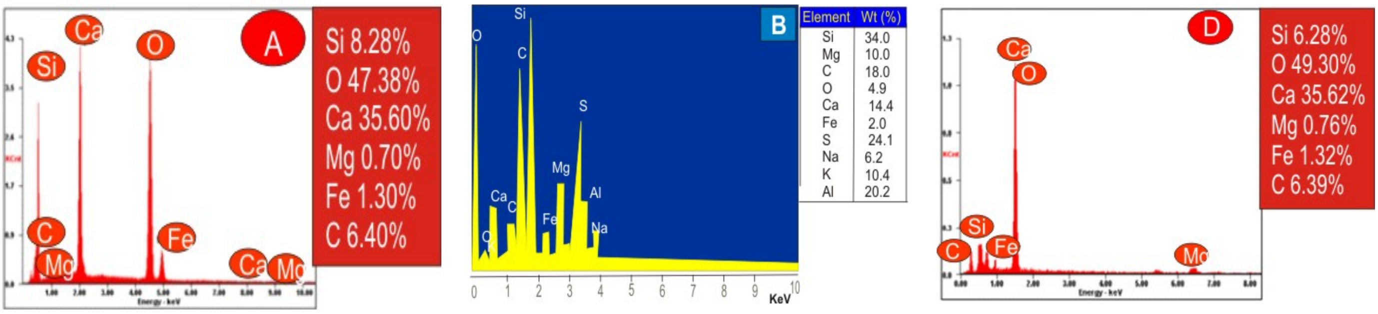
Figure 8 SEM images of mixes A, B and D

3.4.2 Energy Dispersive Spectroscopy (EDS)

It is seen in Figure 9 that mix B contains 34% of silica whereas mixes A and D show 8.28% and 6.28% respectively. Also mix B contains 14.4% of calcium while A and D are 35.60% and 35.62% as well. Only mix B had 20.2% of alumina while the rest had none. This validates the presence of C-S-H in the SEM micrograph for mix B. The selected region in sample B had gel composition of Si/Ca = 2.36 and Al/Ca equivalent to 1.40. While only Si/Ca relationship of 0.23 and 0.18 could be noted for mixes A and D since alumina was absent from their composition. This clearly shows that the presence of C-S-H gel compound in mix B greatly helped in improving the compressive strength much more than the other mixes. A clear difference in the proportion of Si/Ca gel phase among B, A and D could be clearly spotted.

3.4.3 X ray diffraction analysis

The XRD diffraction patterns of two samples A and D are shown in Figures 10 and 11. Sample A has calcite with silica which is the major peak at 17.5^{o} and it is within the amorphous range between 10^{o} and 20^{o} respectively for the 2θ range. This is favourable for pozzolanic activity of the compound. Crystalline compounds such as silica, cristoballite and gismondine are the other major peaks noted at 25.8^{o}, 33^{o} and 47^{o} respectively. However, sample D had major peaks at 27.5^{o}, 35^{o} and 42.5^{o} which belong to crystalline compounds such as calcite and silica. These compounds played major role in the decrease of the compressive strength as seen from the compressive test because of their crystalline forms which originated from the slow rate of cooling of the LBPA within the furnace during the calcining process.



(a) 10% LBPA (b) 15% LBPA (c) 20% LBPA

(a) 10% LBPA (b) 15% LBPA (c) 20% LBPA

Figure 9 Energy Dispersive Spectroscopy

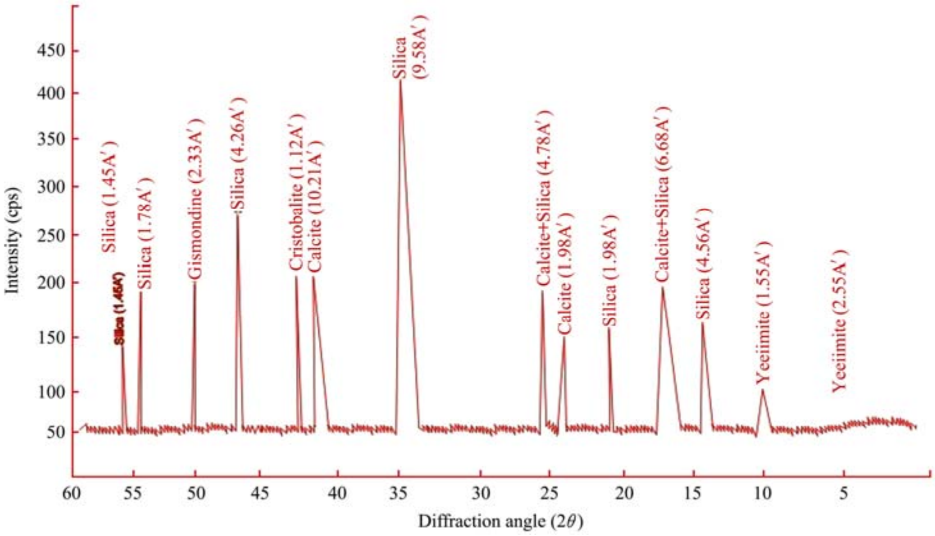


Figure 10 XRD analysis of sample A

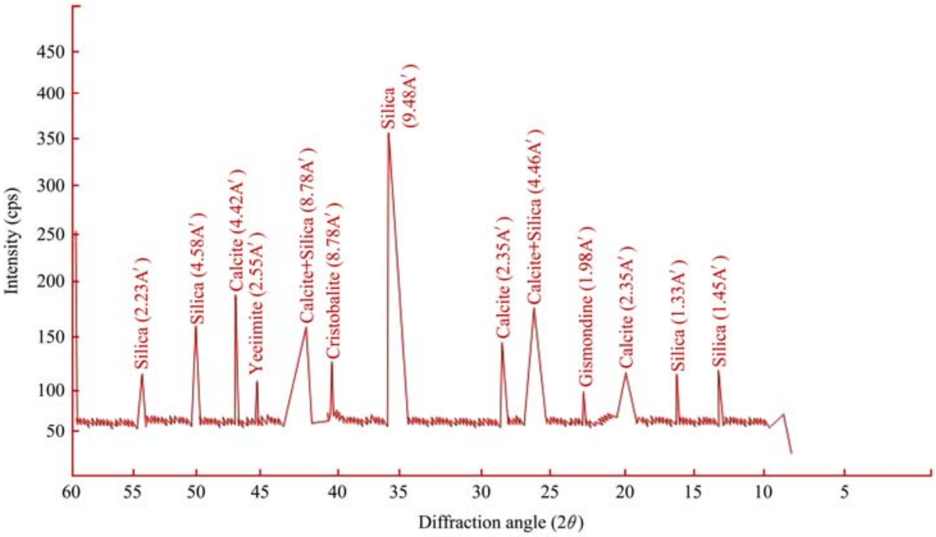


Figure 11 XRD analysis of sample D

4 Conclusion

From the results of the study, the following conclusions are made:

1) Positive effects on all properties were observed on replacement of cement with LBPA as a supplementary cementitious material.

2) Mortar fresh properties results indicated that workability reduces as LBPA increases in the concrete mix. However, increase in both initial and final setting times as the cement binder replacement increases was noted.

3) Increase in LBPA content led to progressive increment in compressive strength up to 15% replacement of cement. But further increase in LBPA content beyond this level led to subsequent decrease in strength because of the lower cement composition and slow hydration process.

4) The obtained compressive strength of this study was within the reported range of other studies conducted on supplementary cementitious materials from agricultural wastes that were used as cement replacement.

5) SEM, EDS and XRD showed pronounced presence of CSH gel in the 15% LBPA replacement of cement more than the other samples which helped to explain the highest compressive strength obtained at the same replacement level. This was also coupled with dense structure with limited presence of voids and pores at the 15% LBPA cement replacement.

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