

Physicochemical Characterisation and Strength-Based Screening of The Calcination Temperature for Locust Bean Pod Ash

^{*1}Manasseh Tyogo, ²Innocent Makoji Bala, ³Akuha Simon Iorshagher

¹Department of Civil Engineering, Joseph Sarwuan Tarka University, Benue State, Nigeria

²Department of Civil Engineering, Nasarawa State University, Keffi, Nigeria

³Department of Civil Engineering, Federal University Otuoke, Bayelsa State, Nigeria

*Corresponding authors' email: manassehtyogo@gmail.com

ABSTRACT

Locust bean pod ash (LBPA) is an abundant West African agricultural residue with potential as a supplementary cementitious material, but the effect of calcination temperature on its composition and performance is poorly documented. Pods were pre-burnt in open air and then calcined in a muffle furnace for 2 hours at 400, 500, 600 and 700 °C. The ashes were characterised by X-ray fluorescence (XRF), X-ray diffraction (XRD) and nitrogen physisorption, and each ash replaced 5% of the cement by mass in concrete with a water-to-binder ratio of 0.58. Calcination raised the $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ sum from 66.99% at 400 °C to 78.30–81.84% at 500–700 °C and lowered the loss on ignition from 5.67 to 3.02%. Carbonate phases matched by XRD at 400–500 °C were no longer leading matches at 600–700 °C, where potassium and calcium-bearing phases were the main matches. The BET surface area was highest at 400 °C (13.38 m²/g) and did not follow the strength ranking, so surface area alone is a poor guide to performance for this ash. The 600 °C ash gave the highest 28-day strength (24.58 N/mm², a relative strength index of 90.67%) and was significantly stronger than the other blends ($p \leq 0.008$). It was 1.68% below the 25 N/mm² design strength and 9.33% below the control, whereas the other blends ranged from 20.03 to 21.49 N/mm². The findings identify 600 °C as the best-performing calcination temperature among the conditions tested, providing a practical basis for optimising LBPA production and assessing its potential as a cost-effective supplementary cementitious material, subject to further validation of strength performance, chemical compliance, and processing economics. Calcination at 600 °C is therefore recommended as the best of the tested temperatures for preparing LBPA.

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INTRODUCTION

Cement production is one of the most carbon-intensive industrial activities, accounting for an estimated 5–8% of global anthropogenic carbon dioxide emissions, and demand continues to grow in rapidly urbanising economies (Gartner & Sui, 2018; Scrivener *et al.*, 2018). A widely used way to reduce the clinker content of concrete is to partially replace Portland cement with supplementary cementitious materials (SCMs). SCMs contribute through a physical filler effect and, where they contain reactive phases, through pozzolanic reactions with the calcium hydroxide released during cement hydration, forming additional calcium silicate hydrate (Lothenbach *et al.*, 2011; Juenger & Siddique, 2015). Conventional SCMs such as coal fly ash, ground granulated blast-furnace slag and silica fume are unevenly available, and in many regions their supply is limited. This has increased interest in locally available agro-industrial ashes (Paris *et al.*, 2016; Juenger *et al.*, 2019).

Ashes from rice husk, corncob, bamboo leaf and other agricultural residues have shown pozzolanic activity when suitably processed and have commonly been used at cement replacement levels of about 5–20% (Aprianti, 2017; Thomas, 2018; Raheem & Ikotun, 2020). Locust bean pod ash is produced from the empty pods of the African locust bean tree (*Parkia biglobosa*), an agricultural residue generated in large quantities across West Africa (Murana, Ochepo, Isah, & Ibedu, 2023). Previous studies have reported pozzolanic

behaviour and acceptable mortar or concrete performance for LBPA at replacement levels between 2.5 and 30%, with the best reported performance usually between about 5 and 15% (Ndububa & Uloko, 2015; Ikumapayi, 2016; Ojewumi *et al.*, 2017; Akpenpuun *et al.*, 2019; Olubajo *et al.*, 2020; Ochola *et al.*, 2021; Isa *et al.*, 2023; Kilani *et al.*, 2025).

The reactivity of a biomass ash depends not only on the replacement level but also on how the ash is produced. The burning temperature, duration and atmosphere control how much organic carbon is removed, the state of the silica and other phases, and the particle and pore structure (Snellings, 2016; Juenger *et al.*, 2019). The thermal behaviour is material specific. Biogenic silica in rice husk is largely non-crystalline and remains so under moderate burning, whereas prolonged or high-temperature burning promotes crystallisation and loss of reactivity; the most reactive rice husk ashes have been obtained at about 500–700 °C (Della *et al.*, 2002; Nair *et al.*, 2008). Similar temperature effects have been reported for sugar cane bagasse ash (Cordeiro *et al.*, 2009), wheat and soybean straw ashes (Šupić *et al.*, 2021) and timber ash (Elinwa & Mahmood, 2002). Recent studies identified 600 °C as the preferred calcination temperature for oil palm kernel shell ash (Arrieta Baldovino *et al.*, 2025) and 700 °C for corncob ash (Okeke *et al.*, 2026). Quartz found in biomass ash is often derived from soil contamination rather than from the plant tissue, and heating at these temperatures does not

convert quartz into reactive amorphous silica (Vassilev *et al.*, 2013).

Most published LBPA studies have used a single, often uncontrolled, burning condition (Isa *et al.*, 2023); for example, Ojewumi *et al.* (2017) ashed the pods at 500 °C for 2 hours. Few studies have examined how calcination temperature affects the composition, crystalline phases, and surface area of LBPA from a single source, as well as the strength of a blended cementitious material made from the same ashes. The present study provides such a same-source comparison at four furnace temperatures. It also shows how far oxide-based screening, surface area and strength agree when ranking the ashes, and what can and cannot be inferred from a four-level screening design.

The aim of this study was to evaluate how the furnace calcination temperature (400, 500, 600 and 700 °C for 2 h), applied to open-air pre-burnt LBPA, affects the properties of the ash and the compressive strength of concrete containing 5% LBPA by mass of cement, and to identify the best-performing tested condition. The objectives were to: (i) determine the physical properties, oxide composition and loss on ignition of the ashes; (ii) identify their crystalline phases by qualitative XRD; (iii) measure their specific surface area and porosity by nitrogen physisorption; and (iv) compare the compressive strength and a study-specific relative strength index of the blended concretes. The scope and limitations are as follows. All ashes came from a single source and were pre-burnt in open air without temperature control before furnace treatment, so the measured differences cannot be attributed solely to furnace temperature. One ash batch was produced at each temperature, XRF and BET were single determinations, and XRD was qualitative. Only one replacement level (5%) was tested; the strength index does not follow ASTM C311, and only the slump and compacting factor were measured on the fresh concrete. The curing water temperature and the loading rate in the strength test were not recorded. With four temperature levels, the study screens the tested conditions; it does not establish a continuous optimum.

MATERIALS AND METHODS

Materials

Ordinary Portland cement (CEM I, Dangote brand) conforming to BS EN 197-1:2011 was used as the binder and stored dry. Locust bean pods were collected as agricultural waste from rural communities in Benue, Nasarawa and Kaduna States, Nigeria. River Benue sand (specific gravity 2.68) was used as the fine aggregate, crushed granite with a 20 mm nominal maximum size (specific gravity 2.70) as the coarse aggregate, and potable water conforming to BS EN 1008:2002 (pH 6.8–8.0) for mixing and curing.

Preparation of Locust Bean Pod Ash

The ash was prepared in two stages. First, the pods were burnt in open air to reduce their bulk volume and combustible content before furnace treatment. The temperature, duration, and oxygen supply during this pre-burning were neither controlled nor recorded. Second, the pre-burnt ash was heated in an electric muffle furnace at the Department of Physics, University of Agriculture, Makurdi (now Joseph Sarwuan Tarka University), for 2 h at 400, 500, 600 or 700 °C. After cooling in the furnace, each ash was ground, passed through a 75 µm (No. 200) sieve, and stored in sealed containers. The ashes are designated LBPA400, LBPA500, LBPA600 and LBPA700. In this paper, “calcination temperature” refers to the furnace set point, and “calcination duration” refers to the 2-hour holding time.

Because the pre-burning was uncontrolled, its peak temperature, duration and atmosphere may have influenced the residual carbon, the chemical composition and the phase assemblage of the starting ash. Local overheating during open burning could also have caused some crystallisation before furnace treatment. The furnace step therefore modified an already partly burnt, possibly variable material, and differences among the four ashes reflect the combined effects of pre-burning and furnace temperature.

Physical Properties

The specific gravity of LBPA and cement was determined by the density bottle method, and fineness was expressed as the mass fraction retained on a 75 µm sieve after dry sieving. Each value is the mean of three determinations.

Chemical Composition

The oxide composition and loss on ignition (LOI) of each ash were determined by X-ray fluorescence (XRF) spectrometry at the Department of Geology, University of Pretoria, South Africa, and those of the cement at the National Geological Centre, Kaduna, Nigeria. Each sample was analysed once. The sum $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ was calculated from the reported oxides. This sum is a bulk chemical quantity that includes both crystalline and non-crystalline forms; it does not measure the reactive fraction. For screening only, the results were compared with the chemical limits of ASTM C618-23e1. That specification covers coal ash and raw or calcined natural pozzolans; biomass ashes fall outside its scope, and ASTM C1709-22 provides a guide for evaluating such alternative SCMs.

Mineralogy

Crystalline phases were identified by X-ray diffraction at the Department of Geology, University of Pretoria, using a Siemens D-500 diffractometer. Patterns were recorded between 5 and 90° 2θ, as shown in Figure 2, and phases were identified by search-match. The analysis was qualitative. Search-match scores and pattern scale factors are not phase mass fractions. No internal standard was added, and no Rietveld quantitative phase analysis was performed, so the amorphous content was not determined.

Specific Surface Area and Porosity

Nitrogen adsorption-desorption isotherms were measured at the University of Pretoria. The specific surface area was calculated using the Brunauer-Emmett-Teller (BET) method (Brunauer *et al.*, 1938), with both multipoint and single-point fits. External surface area was estimated by the t-plot method, and the total pore volume was taken from the amount adsorbed near saturation. The average pore width was calculated from the total pore volume and the BET surface area using Equation 1:

$$d_p = \frac{4000V_p}{S_{\text{BET}}} \quad (1)$$

where d_p is the average pore width (nm), V_p is the total pore volume (cm^3/g), and S_{BET} is the multipoint BET surface area (m^2/g). The factor 4000 combines the factor of 4 for cylindrical pores with the conversion of units to nanometres. The limitations of BET surface area for mesoporous aluminosilicates were considered (López-Pérez *et al.*, 2021), and pore sizes were classified according to the IUPAC (Thommes *et al.*, 2015). Each sample was measured once. BET surface area, sieve fineness and pore size are complementary: the first describes the gas-accessible surface per unit mass, the second the proportion of coarse particles, and the third the internal pore structure.

Concrete Mix Proportions, Specimen Preparation and Compressive Strength

The concrete was proportioned by the absolute volume method for a characteristic 28-day cube strength of 25 N/mm² (target mean strength 31.6 N/mm²), referred to below as the design strength, with a water-to-binder ratio of 0.58 and a design slump of 10–30 mm. The yield equation for one cubic metre is given in Equation 2:

$$\frac{W}{1000} + \frac{C}{1000G_C} + \frac{P}{1000G_P} + \frac{F}{1000G_F} + \frac{K}{1000G_K} + V_a = 1 \quad (2)$$

where W , C , P , F and K are the masses (kg/m³) of water, cement, LBPA, fine aggregate and coarse aggregate; G_C , G_P ,

G_F and G_K are the corresponding specific gravities (dimensionless); 1000 kg/m³ is the density of water; and V_a is the entrapped air volume (m³ per m³ of concrete). The right-hand side, 1, is the volume of concrete being proportioned (1 m³). Specific gravity is the ratio of a material's density to that of water, so each term is a volume in cubic metres. For each ash, 5% of the cement was replaced by an equal mass of LBPA; a control without ash was also made. V_a was set to zero in the design, yielding a binder content of 317.5 kg/m³, with fine and coarse aggregate contents of 2.36 and 3.70 times the binder mass, respectively. The mix proportions are given in Table 1.

Table 1: Mix Proportions of the Concretes (kg/m³)

Mix	Cement	LBPA	Water	Fine agg.	Coarse agg.	Total binder	w/b
Control (0%)	317.50	0	184.15	749.30	1174.75	317.50	0.58
LBPA400 (5%)	301.62	15.88	184.15	749.30	1174.75	317.50	0.58
LBPA500 (5%)	301.62	15.88	184.15	749.30	1174.75	317.50	0.58
LBPA600 (5%)	301.62	15.88	184.15	749.30	1174.75	317.50	0.58
LBPA700 (5%)	301.62	15.88	184.15	749.30	1174.75	317.50	0.58

The LBPA was first blended thoroughly with the cement, and the materials were then mixed at laboratory temperature. For each mix, nine 150 mm cubes were cast, three for each test age of 7, 14 and 28 days (45 cubes in total), in accordance with BS 1881-108:1983, which covers the making of test cubes from fresh concrete. After demoulding, the cubes were cured by full immersion in water until testing. At each age, the cubes were crushed in a compression testing machine, and the compressive strength was calculated as the maximum load divided by the loaded area (22 500 mm²). The value for each mix and age is the mean of three cubes. Slump and compacting factor were measured on the fresh concrete in accordance with BS 1881-102:1983 and BS 1881-103:1983.

Relative Strength Index

A study-specific relative strength index at 5% replacement (RSI₅) was calculated for each age using Equation 3:

$$RSI_5 = 100 \times \frac{A}{B} \quad (3)$$

where RSI₅ is the relative strength index at 5% replacement (%), A is the mean compressive strength of the concrete containing LBPA (N/mm²), and B is the mean compressive strength of the control concrete at the same age (N/mm²). The procedure differs from the strength activity index of ASTM C311/C311M-25, which uses mortar cubes in which 20% of the cement is replaced, with the test mixture water adjusted to give a flow comparable to the control (Bentz *et al.*, 2012). Here, the index was obtained from concrete cubes at 5% replacement and a fixed water-to-binder ratio. RSI₅ is therefore not an ASTM strength activity index and cannot be used to assess conformity with ASTM C618. The 5% level was chosen because earlier LBPA studies reported their best results at low replacement levels (Olubajo *et al.*, 2020; Isa *et al.*, 2023). This supports studying a 5% blend; it does not make the index equivalent to the standard test. Because RSI₅ is calculated directly from strength, it combines dilution, filler, hydration and any pozzolanic effects, and it does not isolate chemical reactivity (Cyr *et al.*, 2006; Snellings & Scrivener, 2016; Li *et al.*, 2018).

Statistical Analysis and Trend Modelling

The strengths at each age are reported as mean and standard deviation (SD). For 28 days, the coefficient of variation (SD as a percentage of the mean) and the 95% confidence interval (CI) for the mean are also provided. The CI was calculated using Equation 4:

$$CI = \bar{x} \pm t_{0.975, n-1} \cdot \frac{s}{\sqrt{n}} \quad (4)$$

where $\bar{x}$ is the mean compressive strength (N/mm²), s is the SD (N/mm²), $n = 3$ is the number of cubes, and $t_{0.975, n-1} = 4.303$ is the two-sided Student's t value for 95% confidence with $n - 1 = 2$ degrees of freedom. Differences between the five mixes at 28 days were tested by one-way analysis of variance (ANOVA). Because the SDs differed by a factor of about 15 (variance ratio about 240), Welch's ANOVA with Games–Howell pairwise comparisons was used as the primary analysis; the classical ANOVA with Tukey comparisons, which assumes equal variances, is reported for comparison. The statistics were computed from the individual cube strengths (three cubes per mix and age) using Python (SciPy). The cubes are specimen replicates from a single ash batch at each temperature, so the tests describe specimen-to-specimen variation; they do not measure variability in ash production. A significance level of 0.05 was used.

To describe trends with calcination temperature T (°C), the general polynomial model in Equation 5 was considered:

$$Y = \beta_0 + \beta_1 T + \beta_2 T^2 + \dots + \beta_k T^k + \varepsilon \quad (5)$$

where Y is the response (28-day strength, oxide sum, LOI or BET surface area), T is the calcination temperature (°C), β_0 to β_k are the least-squares coefficients, k is the polynomial order, and ε is the residual error. With four distinct temperatures, at most four coefficients can be estimated; a cubic model ($k = 3$) passes through every point and leaves no residual degrees of freedom. Linear ($k = 1$, two residual degrees of freedom) and quadratic ($k = 2$, one residual degree of freedom) models were therefore compared using the coefficient of determination (R^2), the adjusted R^2 and the model F-test. For a quadratic model, the stationary point (the temperature at which the fitted response is a maximum or a minimum) was calculated using Equation 6:

$$T^* = -\frac{\beta_1}{2\beta_2} \quad (6)$$

where T^* is the stationary-point temperature (°C), and β_1 and β_2 are the linear and quadratic coefficients of Equation 5. These fits are descriptive only.

RESULTS AND DISCUSSION

Physical Properties of LBPA and Cement

Table 2 gives the physical properties of LBPA and the cement. The specific gravity of LBPA (2.94) was slightly lower than that of the cement (3.02). At a 5% replacement by mass, this difference increases the binder's solid volume by

only about 0.14%, which is negligible. The fraction retained on the 75 μm sieve was 5.6% for LBPA and 2.6% for the cement, so the ash contained a larger proportion of coarse particles. Coarser SCM particles provide less surface for nucleation and reaction, which tends to reduce both the filler effect and the early pozzolanic contribution (Cyr *et al.*, 2006;

Lothenbach *et al.*, 2011). Particle-size effects on reaction kinetics were not measured here, so this is a possible contributor to the strength results in Section 3.5, not a proven cause. The physical properties were measured only on LBPA400, so any change in fineness with calcination temperature is unknown.

Table 2: Physical Properties of LBPA and Cement

Property	LBPA400	OPC
Specific gravity	2.94	3.02
Fraction retained on 75 μm sieve (%)	5.6	2.6

Chemical Composition

Table 3 presents the oxide composition of the cement and the four ashes, and Figure 1 shows the sum of SiO_2 , Al_2O_3 , and Fe_2O_3 , as well as the LOI. SiO_2 was the main oxide in every ash. Its concentration increased from 41.67% at 400 $^\circ\text{C}$ to 52.46% at 700 $^\circ\text{C}$, while Al_2O_3 increased from 14.87% to 17.68%. LOI fell from 5.67 to 3.02%, consistent with progressive removal of residual carbon and other volatile matter. Part of the rise in oxide percentages is a concentration

effect: as combustible and volatile matter is removed, the remaining oxides make up a larger share of the sample. XRF measures total element contents regardless of phase, so a higher SiO_2 percentage does not show that the quantity of silica increased, or that the silica became more amorphous or reactive (Snellings, 2016; Juenger *et al.*, 2019). CaO was about 10% in all ashes, which is typical of plant ashes (Vassilev *et al.*, 2013).

Table 3: Oxide Composition (%) of Cement and LBPA Calcined at Different Temperatures (Single XRF Determinations)

Oxide	OPC	LBPA400	LBPA500	LBPA600	LBPA700
SiO_2	22.35	41.67	49.72	50.65	52.46
Al_2O_3	4.10	14.87	15.11	17.34	17.68
Fe_2O_3	2.00	10.45	13.47	13.85	8.55
CaO	66.23	9.73	10.19	10.01	10.19
MgO	1.82	2.23	3.43	2.26	2.02
K_2O	0.18	4.51	0.57	0.55	0.73
Na_2O	n.r.	1.48	2.09	4.28	3.68
SO_3	2.93	0.16	0.02	0.08	0.31
TiO_2	0.10	0.08	0.14	0.12	0.13
P_2O_5	n.r.	0.04	0.05	0.03	0.07
Other minor oxides	0.13	0.17	0.21	0.49	0.60
LOI	4.00	5.67	4.09	4.01	3.02
Analytical total	103.84	91.06	99.09	103.67	99.44
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	28.45	66.99	78.30	81.84	78.69
$\text{Na}_2\text{O} + 0.658\text{K}_2\text{O}$ (total)	—	4.45	2.47	4.64	4.16

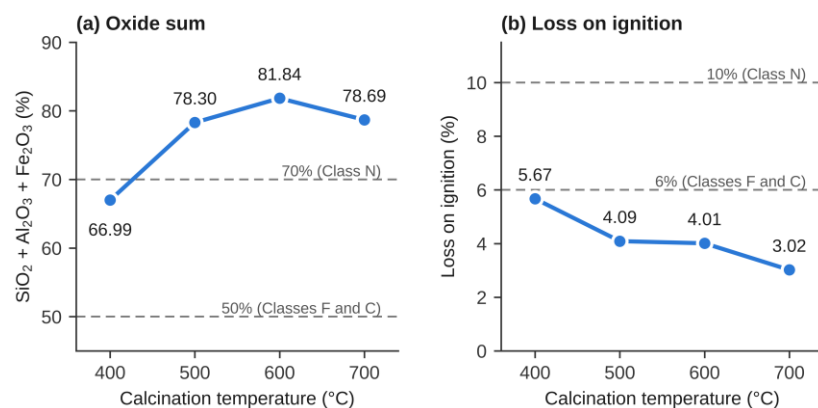


Figure 1: (a) $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ Sum and (b) Loss on Ignition of LBPA Versus Calcination Temperature. Dashed lines show ASTM C618-23e1 Chemical Limits, for Screening Only

The oxide sum increased from 66.99% at 400 $^\circ\text{C}$ to 78.30% at 500 $^\circ\text{C}$ and 81.84% at 600 $^\circ\text{C}$, then fell to 78.69% at 700 $^\circ\text{C}$ (Figure 1a). The 11.31-point rise between 400 and 500 $^\circ\text{C}$ is much larger than the 1.58-point fall in LOI, and it coincided with a fall in K_2O from 4.51 to 0.57%. The fall between 600 and 700 $^\circ\text{C}$ was caused almost entirely by Fe_2O_3 , which

dropped from 13.85 to 8.55%, while $\text{SiO}_2 + \text{Al}_2\text{O}_3$ increased by 2.15 points. Iron oxides are not volatile at these temperatures, and substantial loss of potassium from biomass ash is usually associated with higher temperatures (Vassilev *et al.*, 2013). The irregular changes in K_2O , Na_2O (1.48–4.28%) and Fe_2O_3 are therefore more likely to reflect

compositional differences between the analysed samples, for example, from separate pre-burning or sub-sampling, than a systematic effect of furnace temperature. With single determinations, analytical and sampling variability cannot be estimated. The analytical totals (91.06–103.67% for the ashes and 103.84% for the cement) also fall outside the range normally expected for a complete analysis, and XRD matched phosphate phases (Section 3.3), although XRF found only 0.03–0.07% P_2O_5 . The oxide sum maximum at 600 °C should therefore not be read as a chemical optimum.

For screening, all four ashes exceeded the 50% minimum oxide sum and were below the 18% CaO limit used for Class F in ASTM C618-23e1; the ashes calcined at 500–700 °C also exceeded the 70% minimum for Class N, while LBPA400 (66.99%) did not. All LOI values (3.02–5.67%) were below the 6% limit for Classes F and C and the 10% limit for Class N, and SO_3 ($\leq 0.31\%$) was well below the specification limits (Figure 1). These comparisons are necessary but not sufficient conditions. LBPA is outside the scope of ASTM C618, and the physical requirements (fineness, strength activity index at 20% replacement, water requirement and soundness) and moisture content were not evaluated as specified. No class is therefore assigned. The total alkali content expressed as $Na_2O + 0.658K_2O$ was 2.47–4.64%. This is a total by XRF and is

not equivalent to the available-alkali quantity determined by ASTM C311, so no compliance statement is made; however, the relatively high total alkali content should be considered in further work involving potentially alkali-reactive aggregates.

Mineralogy

Figure 2 shows the XRD patterns of the four ashes, and Table 4 lists the matched phases with their reference codes and search-match scores. The intensity axis uses a square-root scale, which enlarges weak features and compresses strong peaks, so visual comparisons of peak sharpness between patterns should be made with caution. The green line follows the base of each pattern and is taken to be the background fitted by the search-match software; it is not a full-profile phase fit. All four ashes contain crystalline phases. At 400 and 500 °C, the matched phases include quartz (SiO_2) and fairchildite ($K_2Ca(CO_3)_2$), together with langbeinite ($K_2Mg_2(SO_4)_3$) at 400 °C and calcite ($CaCO_3$) at 500 °C. At 600 and 700 °C, carbonates are no longer among the leading matches; the matched phases include potassium calcium phosphate ($KCaPO_4$), calcium silicate (Ca_2SiO_4) and calcium titanate ($Ca_4Ti_3O_{10}$), with potassium sulphate (K_2SO_4), periclase (MgO) and SiO_2 at 600 °C, and augite ($Ca(Fe,Mg)Si_2O_6$) at 700 °C.

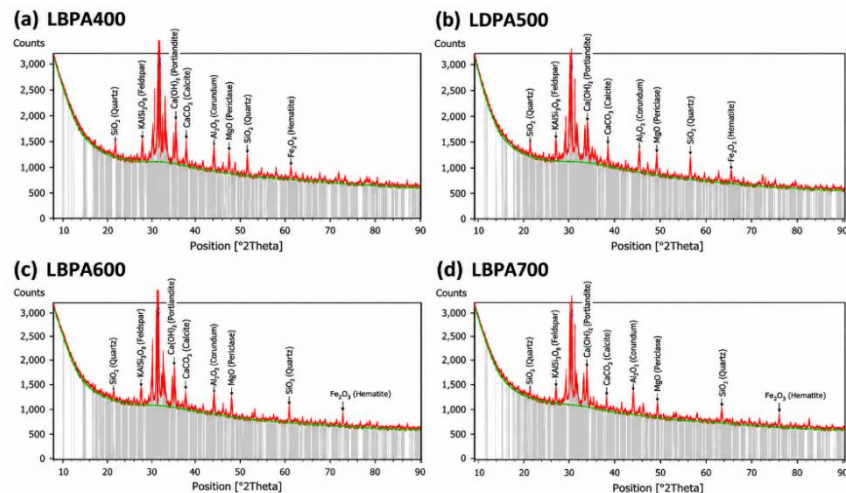


Figure 2: XRD Patterns of LBPA Calcined at (a) 400 °C, (b) 500 °C, (c) 600 °C and (d) 700 °C (Intensity in Counts on a Square-root Scale; Position in 2θ)

Table 4: Phases Matched by XRD Search Match (Reference Code and Score)

Ash	Phase (formula)	Reference code	Score
LBPA400	Quartz (SiO_2)	86-1630	65
	Fairchildite ($K_2Ca(CO_3)_2$)	03-0504	55
	Silicon oxide (SiO_2)	81-0069	55
	Calcium phosphide (Ca_5P_8)*	48-1825	41
	Langbeinite ($K_2Mg_2(SO_4)_3$)	03-0532	38
	Calcium sulphate sulphite ($Ca_3(SO_3)_2SO_4$)	38-0701	38
LBPA500	Quartz (SiO_2)	78-1252	69
	Fairchildite ($K_2Ca(CO_3)_2$)	21-1287	63
	Silicon oxide (SiO_2)	81-0069	51
	Calcium phosphide (CaP)*	16-0728	44
	Calcite ($CaCO_3$)	51-1524	41
	Calcium silicate ($CaSiO_3$)	01-0720	39
	Carbon monoxide (CO)*	76-2378	36
	Aluminium iron silicon ($Al_{0.5}Fe_3Si_{0.5}$)*	45-1205	34
LBPA600	Potassium calcium phosphate ($KCaPO_4$)	33-1002	64
	Periclase (MgO)	45-0946	47
	Potassium sulphate (K_2SO_4)	01-0944	47

Ash	Phase (formula)	Reference code	Score
LBPA700	Calcium sulphide (CaS)	77-2011	41
	Aluminium oxalate (C ₆ Al ₂ O ₁₂)*	37-0488	35
	Calcium silicate (Ca ₂ SiO ₄)	24-0234	35
	Calcium titanate (Ca ₄ Ti ₃ O ₁₀)	14-0152	34
	Silicon oxide (SiO ₂)	80-2148	32
	Potassium calcium phosphate (KCaPO ₄)	33-1002	57
	Potassium carbon sulphide (K ₂ CS ₃)*	20-0937	55
	Calcium phosphide (CaP)*	16-0728	38
	Calcium titanate (Ca ₄ Ti ₃ O ₁₀)	14-0152	34
	Augite (Ca(Fe,Mg)Si ₂ O ₆)	24-0201	32
	Calcium sulphite (CaSO ₃)	04-0599	32
	Calcium silicate (Ca ₂ SiO ₄)	23-1043	28

The loss of carbonate matches between 500 and 600 °C is consistent with the thermal decomposition of calcite and potassium–calcium carbonates reported for biomass ashes (Vassilev *et al.*, 2013). Quartz is crystalline and exhibits little to no pozzolanic reactivity; its presence indicates a nonreactive fraction, possibly derived from soil adhering to the pods. The Ca₂SiO₄ identification at 600–700 °C should be treated as tentative, as the formation of dicalcium silicate via solid-state reaction typically requires higher temperatures, and the match has not been quantitatively confirmed. Ca₂SiO₄ is an anhydrous clinker-type phase, not a hydration product. Several low-score matches, such as the calcium phosphides, carbon monoxide, an aluminium–iron–silicon alloy, aluminium oxalate and potassium carbon sulphide (Table 4), are not plausible in an ash calcined in air and are not interpreted. The potassium calcium phosphate matches are also doubtful, because XRF found only 0.03–0.07% P₂O₅ (Table 3).

The XRD data do not show how much amorphous material each ash contains. All four patterns show a similar steep rise in intensity below about 15° 2θ and an elevated background. At the available image resolution, no systematic change in a broad diffuse band between about 15 and 35° 2θ can be established. Peak sharpness and changes in peak prominence indicate which crystalline phases are present; they do not measure the amorphous fraction. Search-match scale factors

also cannot be converted into phase mass fractions. The amorphous content of each ash, and any change with temperature, therefore, remains unknown. They could be quantified by Rietveld analysis with an internal standard on suitable data (Scrivener *et al.*, 2016). Scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDS) would help describe particle morphology and element distribution, but it cannot, by itself, quantify the bulk amorphous content.

Specific Surface Area and Porosity

Figure 3 shows the multipoint BET surface area and total pore volume. The BET surface area did not decrease steadily. It fell from 13.38 m²/g at 400 °C to 8.60 m²/g at 500 °C, rose to 9.45 m²/g at 600 °C and fell to 6.11 m²/g at 700 °C. The single-point BET area followed the same pattern (11.20, 7.09, 7.49 and 5.00 m²/g). Total pore volume decreased at each step, from 0.0298 to 0.0173, 0.0161 and 0.0122 cm³/g. The average pore width (Equation 1) was 8.9, 8.0, 6.8 and 8.0 nm, which lies in the mesopore range of 2–50 nm (Thommes *et al.*, 2015). This is an average value and does not describe the pore-size distribution. The BJH adsorption average pore diameters (10.0, 9.2, 7.9 and 10.0 nm) were of the same order; the full BJH distributions were not available and are not discussed.

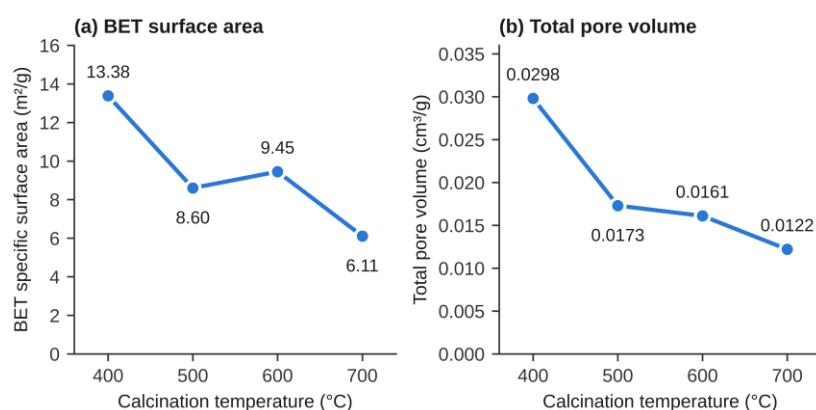


Figure 3: (a) Multipoint BET Specific Surface Area and (b) Total Pore Volume of LBPA Versus Calcination Temperature

The t-plot external surface areas (21.18, 13.66, 15.66, and 10.09 m²/g) exceeded the corresponding BET areas, indicating negative micropore area and volume. A negative fitted micropore quantity has no physical meaning. It indicates that the t-plot model did not fit these essentially non-microporous materials, so the t-plot values are not used

further. The data only indicate that no measurable microporosity was detected.

The general fall in surface area and pore volume is consistent with burning off porous residual char, in line with the falling LOI, and possibly with some particle coarsening at higher temperature. These mechanisms were not verified because neither particle-size distribution nor microscopy was

measured for each ash. The higher BET area at 600 °C than at 500 °C is based on single measurements, so it is not known whether the difference exceeds measurement and sampling variability.

Surface area did not follow the strength ranking. LBPA400 had the largest surface area but the lowest strength (Section 3.5), whereas LBPA600 had the highest strength with an intermediate surface area. In ashes that contain residual carbon, part of the nitrogen-accessible surface belongs to carbon rather than to reactive mineral phases (Juenger *et al.*, 2019). BET surface area is therefore treated here as a supporting characteristic, not as a direct measure of reactivity.

Compressive Strength

The slump of the fresh concrete was 18, 18, 20, 25, and 22 mm, and the compacting factor was 0.90, 0.88, 0.90, 0.94, and

0.89 for the control, LBPA400, LBPA500, LBPA600, and LBPA700 mixes; all slumps were within the 10–30 mm design range. Figure 4 shows the development of compressive strength, and Table 5 summarises the 28-day statistics. The control reached 27.11 N/mm² at 28 days and was the only mix to exceed the 25 N/mm² design strength. None of the blended concretes reached it. Their 28-day strengths were 20.03, 20.77, 24.58, and 21.49 N/mm² for LBPA400, LBPA500, LBPA600, and LBPA700, which are 4.97, 4.23, 0.42, and 3.51 N/mm² (19.88, 16.92, 1.68, and 14.04%) below the design strength. LBPA600 was the strongest blend across all ages, and its 28-day strength was 9.33% lower than the control. Between 7 and 28 days, the control gained 5.63 N/mm² (26.2%), and LBPA600 gained 4.40 N/mm² (21.8%), whereas LBPA400, LBPA500 and LBPA700 gained only 1.21–2.14 N/mm² (6.4–11.1%).

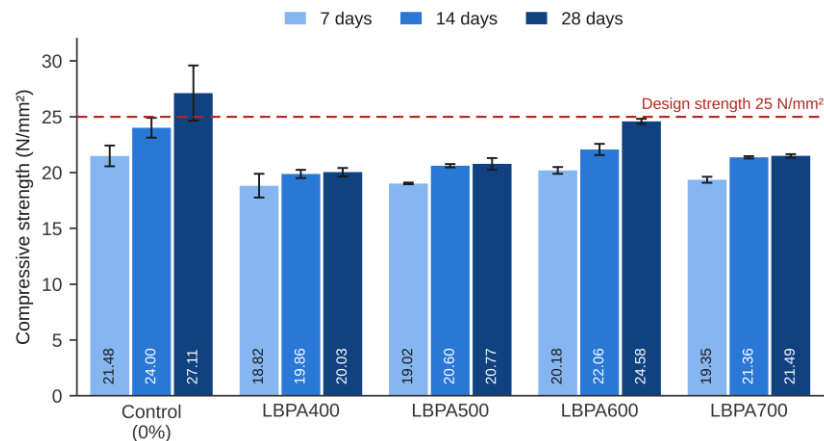


Figure 4: Compressive strength of the control and blended concretes at 7, 14 and 28 days. Bars show the mean of three cubes; error bars show ± 1 SD.

Table 5: Statistical Summary of 28 Day Compressive Strength (n = 3 Cubes Per Mix)

Mix	Mean (N/mm ²)	SD (N/mm ²)	CV (%)	95% CI (N/mm ²)	Difference from 25 N/mm ²
Control (0%)	27.11	2.48	9.1	20.96–33.26	+2.11
LBPA400 (5%)	20.03	0.38	1.9	19.09–20.97	–4.97
LBPA500 (5%)	20.77	0.52	2.5	19.48–22.06	–4.23
LBPA600 (5%)	24.58	0.23	0.9	24.02–25.14	–0.42
LBPA700 (5%)	21.49	0.16	0.7	21.09–21.88	–3.51

Welch's ANOVA showed significant differences among the five mixes ($F(4, 4.69) = 92.2$, $p < 0.001$); the classical ANOVA gave $F(4, 10) = 20.0$, $p < 0.001$. Table 6 gives the pairwise comparisons. With Games–Howell comparisons, LBPA600 was significantly stronger than LBPA400 ($p = 0.001$), LBPA500 ($p = 0.008$) and LBPA700 ($p < 0.001$), and LBPA700 was stronger than LBPA400 ($p = 0.043$). None of the differences between the control and the blends was significant ($p = 0.11$ – 0.55). This is because the control had a large SD (2.48 N/mm²), so the comparisons have very few effective degrees of freedom and low power. A non-significant result does not show that LBPA600 matched the control; neither a difference nor equivalence was shown.

When equal variances were assumed (Tukey), the control was stronger than LBPA400, LBPA500, and LBPA700 ($p \leq 0.001$), but not LBPA600 ($p = 0.125$), and the difference between LBPA600 and LBPA700 was only just significant ($p = 0.0499$). Conclusions involving the control, therefore, depend on the variance assumption. The ranking of LBPA600 above the other blends holds under both analyses, although its margin over LBPA700 is borderline under the Tukey test. Because each mix came from a single ash batch, these results compare specimens rather than independently reproduced calcination processes.

Table 6: Pairwise Comparisons of 28 Day Compressive Strength

Comparison	Difference (N/mm ²)	Games–Howell p	Tukey p
Control – LBPA400	7.08	0.109	< 0.001
Control – LBPA500	6.34	0.131	< 0.001
Control – LBPA600	2.53	0.548	0.125
Control – LBPA700	5.62	0.171	0.001
LBPA600 – LBPA400	4.55	0.001	0.005
LBPA600 – LBPA500	3.81	0.008	0.015
LBPA600 – LBPA700	3.09	< 0.001	0.0499
LBPA700 – LBPA400	1.46	0.043	0.556
LBPA700 – LBPA500	0.71	0.378	0.937
LBPA500 – LBPA400	0.74	0.407	0.927

Relative Strength Index

Figure 5 shows RSI₅ at each age, calculated from the mean strengths in Figure 4. At 28 days, RSI₅ was 73.88%, 76.63%, 90.67%, and 79.26% for LBPA400, LBPA500, LBPA600, and LBPA700. RSI₅ decreased with age for every ash,

because the blends gained strength more slowly than the control. The decrease was smallest for LBPA600 (from 93.95 to 90.67%). At 7 days, both LBPA600 (93.95%) and LBPA700 (90.08%) exceeded 90%.

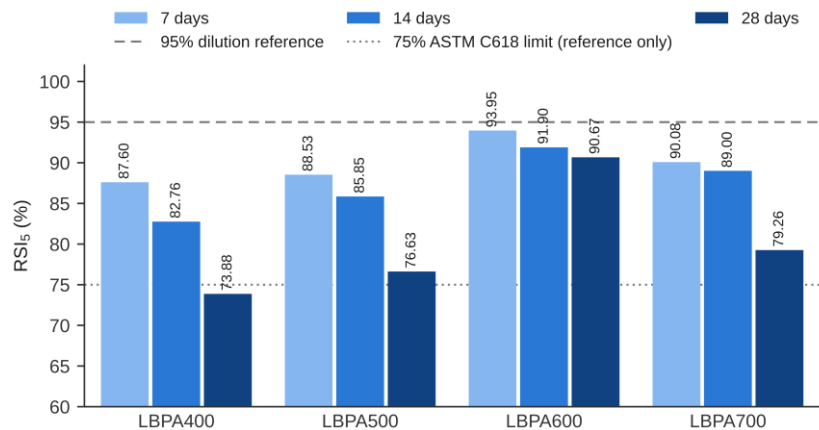


Figure 5: Relative Strength Index (RSI₅) of Concretes Containing 5% LBPA at 7, 14 and 28 days. The Dashed Line at 95% Marks the Cement Fraction Remaining at a 5% Replacement; the Dotted Line at 75% is the ASTM C618 Strength Activity Limit, shown for Reference only, as RSI₅ is not the ASTM C311 Index

A useful reference is dilution. Replacing 5% of the cement with an inert material at the same water-to-binder ratio leaves 95% of the cement and raises the effective water-to-cement ratio from 0.58 to about 0.61, so an index near or somewhat below 95% would be expected with no net contribution from the ash. LBPA600 (90.67%) was close to this reference, whereas LBPA400, LBPA500 and LBPA700 (73.88–79.26%) were well below it. The data, therefore, do not show a net positive (filler or pozzolanic) contribution from any ash by 28 days. They suggest that LBPA400, LBPA500, and LBPA700 reduced strength by more than dilution alone would. Possible reasons include residual carbon, coarser particles, alkali and sulphate content and batch differences, but these were not investigated and remain hypotheses. RSI₅ is calculated directly from strength, so it reproduces the strength ranking exactly. Its agreement with the strength

results is therefore not independent confirmation of reactivity. The ranking of RSI₅ also does not follow the oxide sum: LBPA500 and LBPA700 had oxide sums similar to LBPA600 (78.30% and 78.69% against 81.84%) but much lower RSI₅. Direct reactivity tests, such as the R3 heat-release or bound-water tests, would be needed to separate chemical reactivity from physical effects (Li *et al.*, 2018; Snellings *et al.*, 2019).

Trend Modelling and Best-performing Tested Condition

Table 7 compares linear and quadratic fits of 28-day strength, oxide sum, LOI and BET surface area against calcination temperature. RSI₅ is not fitted separately: it equals strength multiplied by a constant (100/27.11), so its model has identical R², p-value and stationary point and would count the same data twice.

Table 7: Polynomial Trend Models Fitted to the four Calcination Temperatures (T in °C)

Response	Order	Fitted equation	R ²	Adj. R ²	p	Residual df
28-d strength (N/mm ²)	1	$17.22 + 8.18 \times 10^{-3}T$	0.279	-0.082	0.472	2
	2	$-10.60 + 0.1137T - 9.59 \times 10^{-5}T^2$	0.586	-0.241	0.643	1
Oxide sum (%)	1	$55.20 + 3.864 \times 10^{-2}T$	0.588	0.382	0.233	2
	2	$-49.63 + 0.4363T - 3.615 \times 10^{-4}T^2$	1.000	0.999	0.021	1
LOI (%)	1	$8.614 - 8.03 \times 10^{-3}T$	0.895	0.843	0.054	2
	2	$12.89 - 2.426 \times 10^{-2}T + 1.475 \times 10^{-5}T^2$	0.919	0.758	0.284	1
BET area (m ² /g)	1	$20.91 - 2.096 \times 10^{-2}T$	0.804	0.707	0.103	2
	2	$31.35 - 6.056 \times 10^{-2}T + 3.60 \times 10^{-5}T^2$	0.823	0.470	0.420	1

The strength data follow neither model well. The quadratic fit has $R^2 = 0.59$, a negative adjusted R^2 (-0.24) and $p = 0.64$, so it explains no more of the variation than the mean once its extra term is allowed for. Its stationary point (593 °C) is therefore not a reliable estimate of an optimum. The near-exact quadratic fit of the oxide sum ($R^2 = 1.00$, $p = 0.021$) has only one residual degree of freedom, and its curvature is controlled by the low Fe_2O_3 value at 700 °C (Section 3.2), so its stationary point (603 °C) is also not evidence of an optimum. LOI is described adequately by a linear decrease ($R^2 = 0.90$), and the quadratic term adds little. Alternative forms, such as exponential decay for LOI, cannot be distinguished reliably with four points.

Three different quantities have been associated with “about 600 °C” and must not be used interchangeably: the best-performing tested temperature (600 °C, a measured result), the stationary point of the strength fit (593 °C, from a non-significant model) and the stationary point of the oxide-sum fit (603 °C, a chemical quantity). The original submission also reported a desirability peak near 615 °C and an optimum range of 600–620 °C. That analysis has been withdrawn because its desirability limits and weights were not defined, it counted strength twice (both strength and its derived index), and the four-level data cannot support a continuous optimum. An ANOVA across the tested levels also cannot show where the optimum lies among them. BET surface area was not used as an optimisation objective because no defensible target exists for it: the largest area was observed in the ash with the most residual carbon and the lowest strength. It is retained as a supporting measurement.

The supported finding is that 600 °C was the best-performing of the four tested calcination temperatures for concrete strength at 5% replacement. The true optimum may lie anywhere between about 500 and 700 °C and can only be located by new, replicated experiments.

CONCLUSION

The following conclusions apply to LBPA prepared by uncontrolled open-air pre-burning followed by 2 h of furnace treatment, and to concrete with 5% LBPA by mass of cement:

- The $SiO_2 + Al_2O_3 + Fe_2O_3$ sum was 66.99, 78.30, 81.84 and 78.69% at 400, 500, 600 and 700 °C, and LOI decreased from 5.67 to 3.02%. These were single determinations with incomplete analytical totals (91.06–103.67%). The ashes calcined at 500–700 °C exceeded the 70% oxide-sum threshold of ASTM C618-23e1 Class N, but no class or compliance was established.
- Qualitative XRD showed crystalline assemblages in all ashes. Quartz and fairchildite were matched at 400 and 500 °C, with calcite at 500 °C; at 600–700 °C, carbonates were no longer leading matches, and

potassium- and calcium-bearing phases were matched. The phosphate matches are doubtful given the low P_2O_5 content ($\leq 0.07\%$). The amorphous content was not determined, and no increase in amorphous silica was shown.

- The BET surface area was 13.38, 8.60, 9.45 and 6.11 m²/g and the total pore volume fell from 0.0298 to 0.0122 cm³/g. Surface area did not rank the ashes in the same order as strength.
- The 28-day strengths of the blends (20.03, 20.77, 24.58 and 21.49 N/mm²) were all below the 25 N/mm² design strength. The strongest blend, LBPA600, was 0.42 N/mm² (1.68%) below the design strength and 9.33% below the control (27.11 N/mm²).
- LBPA600 was significantly stronger than the other blends (Games–Howell, $p \leq 0.008$). Its difference from the control was not statistically resolved ($p = 0.55$), so neither a difference nor equivalence was shown.
- The 28-day relative strength index (RSI₅) was 73.88, 76.63, 90.67 and 79.26%. All values were below the 95% dilution reference, and RSI₅ is not an ASTM C311 strength activity index.
- Quadratic models of strength against temperature were weak ($R^2 = 0.59$, adjusted $R^2 = -0.24$, $p = 0.64$) and did not locate an optimum. Of the conditions tested, 600 °C was the best-performing calcination temperature.

RECOMMENDATIONS

- Calcine homogenised raw pods directly in a controlled furnace, without open-air pre-burning, and produce at least three independent ash batches per temperature.
- Test additional temperatures between 500 and 700 °C and confirm the best condition experimentally. If a response-surface model is wanted, use a designed experiment in calcination temperature and duration, for example, a two-factor central composite design centred on 600 °C and 120 min with replicated centre points.
- Quantify the amorphous content by Rietveld analysis with an internal standard, use SEM/EDS for morphology, and apply a direct reactivity test such as R3.
- Determine the ASTM C311 strength activity index at 20% replacement, together with water requirement and setting behaviour, replicate XRF and BET measurements, and durability, including alkali–silica reaction in view of the total alkali content.

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