

Article

Effect of Activator-to-Binder Ratio and Curing Temperature on the Properties of Low-Density Alkali-Activated Mortar Incorporating Phragmites Australis Ash

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Abstract

Valorizing agricultural residues as cement-free binders may reduce dependence on Portland cement. However, the performance of silica-rich biomass ashes under alkali activation remains insufficiently understood. This study explores the effects of alkali activator-to-binder ratio (A/B), curing temperature, and age on mortars containing *Phragmites australis* ash (PAA) as the sole binder. Specimens with A/B ratios of 1.0, 1.1, and 1.2 were activated with sodium silicate and 12 M sodium hydroxide (SS/SH ratio of 2.5) and cured at 20, 40, and 80 °C. Flow, density, ultrasonic pulse velocity, flexural strength, compressive strength, and dimensional stability were evaluated. Compared with the Portland-cement control, PAA mortars showed lower flow and mechanical performance, but densities remained below 1500 kg/m³ under all curing conditions. Increasing A/B significantly increased flexural and compressive strengths at 28 days. Elevated-temperature curing significantly increased ultrasonic pulse velocity and both strengths. The maximum PAA compressive strength was 4.67 MPa at 90 days after curing at 80 °C, representing a reduction of 62%. PAA mortars showed higher drying and autogenous shrinkage with increases of 8.5–9.5% and 2.9–4.4%, respectively. However, PAA mixes resulted in lower chemical shrinkage than the control mix, by around 0.2–0.6%. The present results suggest that PAA can be used as a low-density non-structural mortar in non-load-bearing applications, subject to durability and component-scale studies.

Keywords: alkali-activated mortar; biomass ash; activator-to-binder ratio; curing temperature; low-density mortar; dimensional stability



Academic Editor: Alessio Cascardi

Received: 26 August 2026

Revised: 26 September 2026

Accepted: 29 September 2026

Published: 30 September 2026

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1. Introduction

The construction sector increasingly faces challenges in reducing its environmental impact worldwide while preserving the mechanical properties, durability, sustainability, and serviceability of construction materials. The construction industry accounts for nearly 32% of global energy consumption and around 34% of carbon dioxide emissions [1]. Concrete remains the dominant material used worldwide in construction, due to its versatility, durability, availability, and excellent properties [2,3]. However, relying on Portland cement (PC) as the principal binder used in concrete poses serious environmental concerns, since

the production of cement is associated with nearly 8% of greenhouse gas emissions, high energy consumption, and release of carbon dioxide emissions [4]. So, developing substitutes and alternatives to cementitious binders and systems that are capable of decreasing the environmental burden associated with conventional concrete while maintaining good properties has become a vital research concern [5,6].

Of particular interest among cement-free binders are alkali-activated materials (AAMs), as various industrial and agricultural wastes may be used as precursor materials. Geopolymers are often considered a subclass of AAMs, assuming alkali activation results in a 3D aluminosilicate structure. Thus, research into geopolymers offers insight into the effects of precursors, activators, and curing conditions on alkali-activated binders. Precursors frequently used in literature consist of fly ash, municipal solid-waste ash, metakaolin, silica fume, ground-granulated blast-furnace slag, rice husk ash, and sugarcane bagasse ash [7–13]. Unlike Portland cement concrete, which undergoes a hydration reaction, a geopolymer undergoes a geopolymerization reaction in which material with a high silicate content dissolves in an alkaline environment to form a three-dimensional aluminosilicate network through three stages: dissolution, crystallization, and polycondensation [14–16]. This provides an opportunity to incorporate agricultural wastes into eco-friendly and sustainable construction materials, thereby contributing to a reduced carbon footprint [17,18].

Unlike cement-based concrete that has standard properties, geopolymer concrete depends heavily on the constituents of the materials and the processing conditions [19]. Geopolymer concrete properties rely on the chemical composition, physical characterization, activators chosen, concentration of sodium hydroxide (SH), ratio of sodium silicate to sodium hydroxide (SS/SH), alkaline-to-binder ratio (A/B), curing duration, and curing temperature [15,20–27]. These various parameters influence the geopolymerization process and development of the binder and consequently affect the fresh, mechanical, and durability properties of concrete and mortar.

The effect of curing conditions represents a significant consideration in the development of the geopolymerization reaction. Elevated temperatures (40 °C and above) are usually adopted for geopolymer concrete because thermal curing can accelerate the geopolymerization reaction and help develop early strength [28]. However, the use of ambient curing temperature has also been explored, despite the challenges associated with strength development, dimensional stability, and long-term durability [29].

Previous studies have investigated the use of various materials to totally replace cement in concrete and mortar. The use of 15% of rice husk ash (RHA) with ground granulated blast furnace slag (GGBFS) increased compressive and tensile strength and lowered chloride permeability and sorptivity [30]. This was related to the high $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio and the high fineness of RHA that led to increased performance and strength [30]. The density of geopolymer mixes containing rice husk ash ranges from 1.2 g/cm³ to 1.7 g/cm³ [31]. Also, geopolymer mixes containing waste clay bricks, fly ash, and slag showed higher drying shrinkage than cement-based concrete, as geopolymer mixes are considered to be highly sensitive to moisture variation [32].

Limited studies have been conducted on the use of agricultural waste materials to form geopolymer mixes. Accordingly, further investigation of the use of bio-ash materials to totally replace cement in concrete and mortar is needed. This paper examines the effect of different activator-to-binder ratios on the performance of alkali-activated PAA mortar mixes containing *Phragmites australis* ash (PAA) as a total cement replacement. The reported experimental program assessed the flow, density, ultrasonic pulse velocity (UPV), flexural strength, compressive strength, drying shrinkage, autogenous shrinkage, and chemical shrinkage at various curing temperatures and ages in comparison to control mixes.

2. Materials and Methods

2.1. Materials and Mix Proportions

The sand used consisted of particles that were passed through a 4.75 mm sieve and retained on a No. 200 sieve, with a specific gravity of 2.45. Portland cement conforming to EN 197-1 was used in the control mixtures [33]. It consisted of 18.10% SiO₂, 61.55% CaO, and 4.29% Al₂O₃, while the remaining constituents, including Fe₂O₃, K₂O, MgO, SO₃, Na₂O, TiO₂, MnO, P₂O₅, Cr₂O₃, and Cl, collectively accounted for 16.06%. In the alkali-activated mixes, *Phragmites australis* ash (PAA) was incorporated as a total cement replacement. The PAA was collected, dried at 28 °C under sunlight, and burned in a totally closed container for 3 h at approximately 350 °C without the use of fuel; this can be considered a relatively low combustion temperature in order to reduce energy consumption [34]. The ash was then ground using a Los Angeles machine, and sieved through a 300 µm sieve. The PAA consisted of 76.15% SiO₂, 6.15% CaO, and 0.84% Al₂O₃; the remaining components, including Fe₂O₃, K₂O, MgO, SO₃, Na₂O, TiO₂, MnO, P₂O₅, Cr₂O₃, Cl, collectively accounted for 16.86%. Fourier transform infrared spectroscopy (FTIR) was carried out to assess the chemical bonds present in the *Phragmites australis* ash (PAA). There appeared to be several chemical bonds including Si-O, Al-O, H-O-H, O-H, and C=O; Si-O was present in a high quantity, as previously reported [35]. The specific gravity and LOI of the cement and PAA were 3.00 and 0.88, and 7.81% and 18%, respectively. The particle size distribution of both PAA and cement is shown in Figure 1. Regarding alkaline activators, sodium silicate, Na₂SiO₃ (SS), with an SiO₂/Na₂O ratio equal to 3.3 ± 0.1 , and sodium hydroxide, NaOH (SH), with a concentration of 12 M and an SS/SH ratio of 2.5, were prepared 24 h before casting and used in the alkali-activated mixtures. Al/B ratios of 1.0, 1.1, and 1.2 were used, and the corresponding PAA mixtures are designated PAA_1, PAA_1.1, and PAA_1.2, where Al is considered as the combination between SS and SH based on the ratios adopted. Tap water was used for the control mixes, designated by PC, with a fixed w/c ratio of 0.5. The binder-to-sand ratio was fixed at 1:3. Table 1 presents the different ratios used, and Table 2 presents the quantities of the mortar mixes.

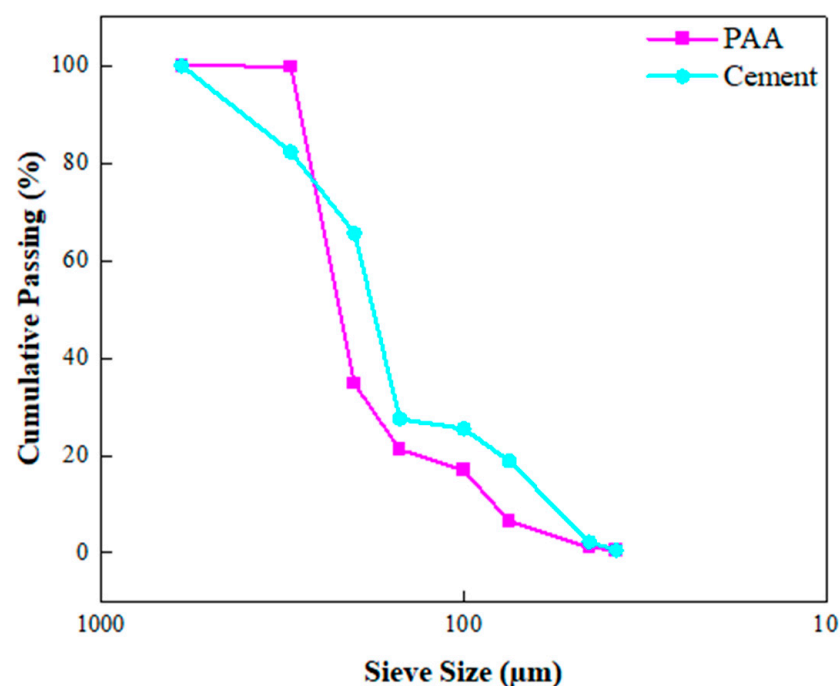


Figure 1. Particle Size Distribution for PAA and Cement.

Table 1. Ratios for Various Mortar Mixes.

Mix ID	PAA	PC	Sand	SS/SH	Al/B	W/C	NaOH (M)
PAA_1	1	0	3	2.5	1	0	12
PAA_1.1	1	0	3	2.5	1.1	0	12
PAA_1.2	1	0	3	2.5	1.2	0	12
PC	0	1	3	0	0	0.5	0

Table 2. Quantities for Various Mortar Mixes (kg/m³).

Mix ID	PAA	PC	Sand	SH	SS	Water
PAA_1	321.32	0.00	963.95	91.80	229.51	0.00
PAA_1.1	313.74	0.00	941.22	98.60	246.51	0.00
PAA_1.2	306.52	0.00	919.55	105.09	262.73	0.00
PC	0.00	485.95	1457.85	0.00	0.00	242.98

2.2. Specimen Preparation, Casting and Curing

Sand and PAA were mixed in a mixer; the alkali activator was then added gradually. For the control mixture, sand and cement were mixed first, after which water was added gradually until a homogeneous mixture was obtained. For the control mix, two curing regimes were applied: water curing, designated PC_W, and temperature curing at ambient temperature, 40 °C, or 80 °C, designated PC_T. For analysis of mechanical properties, mortar prisms with dimensions of 40 × 40 × 160 mm were cast and tested after 7, 28, and 90 days of curing. PAA and PC_T specimens assigned to thermal curing were kept in an oven at 40 or 80 °C for 48 h from casting and were then removed from oven and kept in plastic bags until testing. Specimens cured under ambient conditions (20 °C) were directly covered with plastic bags prior casting and stored until testing. Regarding the PC_W mix, specimens were immersed in water until testing. For dimensional-stability tests, prisms with dimensions of 25 × 25 × 285 mm were cast and tested after 1, 7, 28, and 90 days, with day 1 defined as the day following the 48 h curing period. Alkali-activated specimens were cured at 80 °C, and PC mixtures were divided into two sets: one cured at 80 °C and designated PC_T, and the other cured under normal conditions and designated PC_N. The chemical-shrinkage testing was performed after 1, 7, 28, and 90 days. For each variable, the results from three specimens were averaged.

2.3. Testing

Different tests were performed on several mortar mixtures, including the alkali-activated and control specimens. Flow testing was conducted using a flow table, by applying 25 drops and measuring the diameter of flow of the mortar to check fresh properties. Density, UPV, flexural strength, and compressive strength were measured to evaluate the hardened properties where the density testing was performed to measure the bulk density of the hardened mortar. Drying, autogenous, and chemical shrinkage tests, including measurements of mass loss and volume change, were performed to assess dimensional stability. Table 3 presents the tests conducted, together with the corresponding standards and equations.

Table 3. Tests, Standards, and Formulas.

Testing	ASTM Standard	Equations
Flow	ASTM C1437 [36]	$F = \frac{D_{avg} - D_0}{D_0} \times 100 \quad (1)$ $F: \text{flow (\%)}$ $D_{avg}: \text{average diameter in opposite directions (cm)}$ $D_0: \text{diameter of the mold (cm)}$
Density	ASTM C642 [37]	$\rho = \frac{m}{V} \quad (2)$ $\rho: \text{density (kg/m}^3\text{)}$ $m: \text{mass (kg)}$ $V: \text{volume of specimen (m}^3\text{)}$
UPV	ASTM C597 [38]	$UPV = \frac{d}{t} \quad (3)$ $UPV: \text{ultrasonic pulse velocity (km/s)}$ $d: \text{distance between transducers (km)}$ $t: \text{time (s)}$
Flexural Strength	ASTM C348 [39]	$\sigma = \frac{3PL}{2bd^2} \quad (4)$ $\sigma: \text{flexural strength (MPa)}$ $P: \text{load (N)}$ $L: \text{length of the prism (mm)}$ $b: \text{width of the prism (mm)}$ $d: \text{depth of the prism (mm)}$
Compressive Strength	ASTM C349 [40]	$f'_c = \frac{P}{A} \quad (5)$ $f'_c: \text{compressive strength (MPa)}$ $P: \text{load (N)}$ $A: \text{cross-sectional area (mm}^2\text{)}$
Drying and Autogenous Shrinkage	ASTM C157 [41]/ASTM C1698 [42]	$\varepsilon = \frac{L_i - L_t}{L_i} \quad (6)$ $\varepsilon: \text{shrinkage (\%)}$ $L_t: \text{length at different time intervals (mm)}$ $L_i: \text{initial length (mm)}$
Mass Loss	-	$\Delta m = \left(\frac{m_i - m_t}{m_i} \right) \times 100 \quad (7)$ $\Delta m: \text{mass loss (\%)}$ $m_t: \text{mass at different time intervals (g)}$ $m_i: \text{initial mass (g)}$
Volume Change	-	$\Delta V = \left(\frac{V_i - V_t}{V_i} \right) \times 100 \quad (8)$ $\Delta V: \text{change in volume (\%)}$ $V_t: \text{volume at different time intervals (m}^3\text{)}$ $V_i: \text{initial volume (m}^3\text{)}$
Chemical Shrinkage	ASTM C1608 [43]	$CS = \frac{\Delta V}{M} \quad (9)$ $CS: \text{chemical shrinkage (mL/g)}$ $\Delta V: \text{change in water level (mL)}$ $M: \text{mass of the mortar (g)}$

3. Results and Discussion

3.1. Flow

Figure 2 displays the effect of the Al/B ratios on the flow of alkali-activated mortar mixtures compared with the control mix. Results show that all alkali-activated mixes, despite the Al/B used, recorded lower flow than the control mix. This may be attributed to the porous nature of PAA, which increases water absorption and consequently reduces mortar flow [44]. Comparing alkali-activated mixes with different Al/B ratios, as Al/B

increased, the flow became higher. This is related to the higher activator liquid content that led to the increase in the flow of the alkali-activated mixes [45].

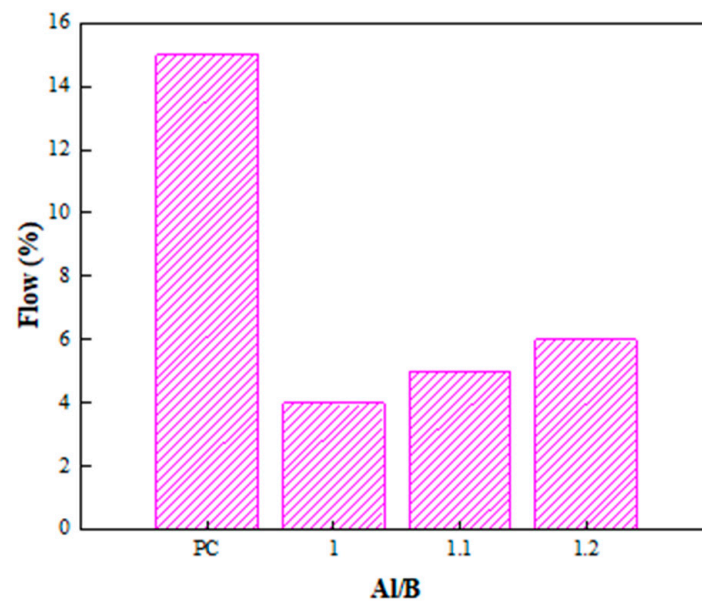


Figure 2. Flow of Mortar with Different Al/B Ratios Compared to Control Mix.

3.2. Density

Figure 3 shows the density of PAA mixes with different Al/B ratios at various curing ages and temperatures compared with the control mixture. These results show that the density of alkali-activated mixes at all tested conditions was lower than that of the control mix. For example, PAA mixes with an Al/B ratio of 1.2 cured at 40 °C and at 28 days had a density of 1470.12 kg/m³ while the control mix recorded a value of 2197.46 kg/m³, representing a reduction of 33%. This may be related to the lower specific gravity of PAA in comparison to the specific gravity of cement [46]. For all tested mixes in all conditions, the density of mortar increased with the increase in curing age. As an example, the density of PC_80 mix varied from 2059.18 kg/m³ to 2079.69 kg/m³ as the time was extended from 7 to 90 days. As the time passed, the reaction was improved, leading to enhancement in the packing effect in the internal matrix and resulting in an improvement in the density [47]. The increase in Al/B ratio was accompanied by an increase in the Si content, which led to enhancement of the matrix by improving the Si-O-Si bond, resulting in a denser structure with higher density [48]. So, the density increased as the Al/B ratio became higher. For example, at ambient temperature and 7 days, the PAA mixture with an Al/B ratio of 1.0 had a density of 1316.41 kg/m³, whereas the mixture with an Al/B ratio of 1.2 had a density of 1400.39 kg/m³, representing an increase of 6.4%. Regarding the effect of curing temperatures on the density of PAA mixes, mixes cured at 40 °C had the highest density values, since a balanced behavior between retention of moisture and the alkali-activation process was achieved at moderate curing temperatures. However, at high temperatures, water may escape, leading to a decrease in the density [49]. It is also possible that there was a difference in phase changes between curing at 40 °C and 80 °C.

3.3. Ultrasonic Pulse Velocity (UPV)

Figure 4 demonstrates the variation of UPV for PAA mixes with various Al/B ratios at different curing ages and temperatures relative to the control mix. Comparing the control mix with PAA mixes, the UPVs of the PC mixes were much higher than those of the alkali-activated mixes. To illustrate, for PAA mix with Al/B equal to 1.2, at 80 °C and 90 days of

curing, the UPV value was 1.88 km/s, while the UPV of the control mix was 2.99 km/s. This indicates a reduction of 36% between the PAA and control mix. This may be related to the porous structure of the alkali-activated mortar mixes requiring more time for the pulse to travel, leading to lower UPV values [50]. Regarding the effect of curing age on UPV values, the UPV increased in all the tested mixes as time passed, regardless of the conditions. For example, for PC_40, the UPV value varied from 3.07 km/s to 3.23 km/s as the times extended from 7 days to 90 days. This may have been due to the continuous hydration and alkali-activation reactions over time, leading to the filling of pores in the matrix and resulting in an increase in UPV values [51]. The UPV increased with the variation in Al/B ratio from 1.0 to 1.2 in increments of 0.1, due to the formation of more alkali-activation reaction products leading to less porosity where voids were filled more [7]. So, for PAA mixes at 80 °C and 28 days of curing, the UPV increased from 1.52 km/s at Al/B equal to 1 to 1.88 km/s for the mix with Al/B ratio of 1.2, representing an increase of 23.7%. UPV was enhanced as the curing temperature increased. This may be attributed to facilitated gel formation within the specimens, which enhanced the UPV [26]. As an example, at ambient curing and at 90 days, the mix with 1.2 Al/B ratio recorded a value of 0.87 km/s and increased to 1.88 km/s when the temperature was 80 °C.

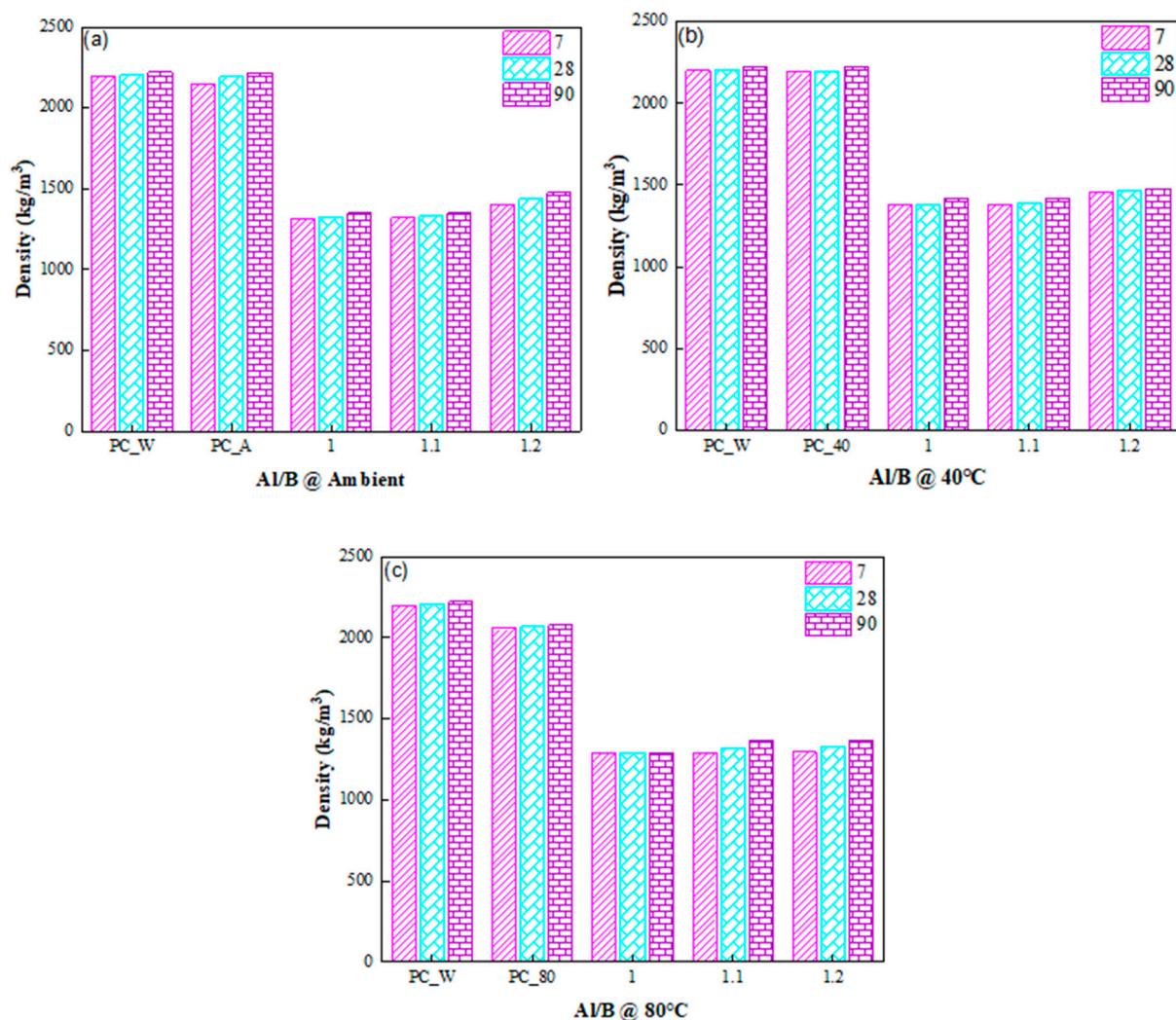


Figure 3. Density of Mortar with Different Al/B Ratios Compared to Control Mix at Various Testing Ages and Temperatures: (a) Ambient, (b) 40 °C, and (c) 80 °C.

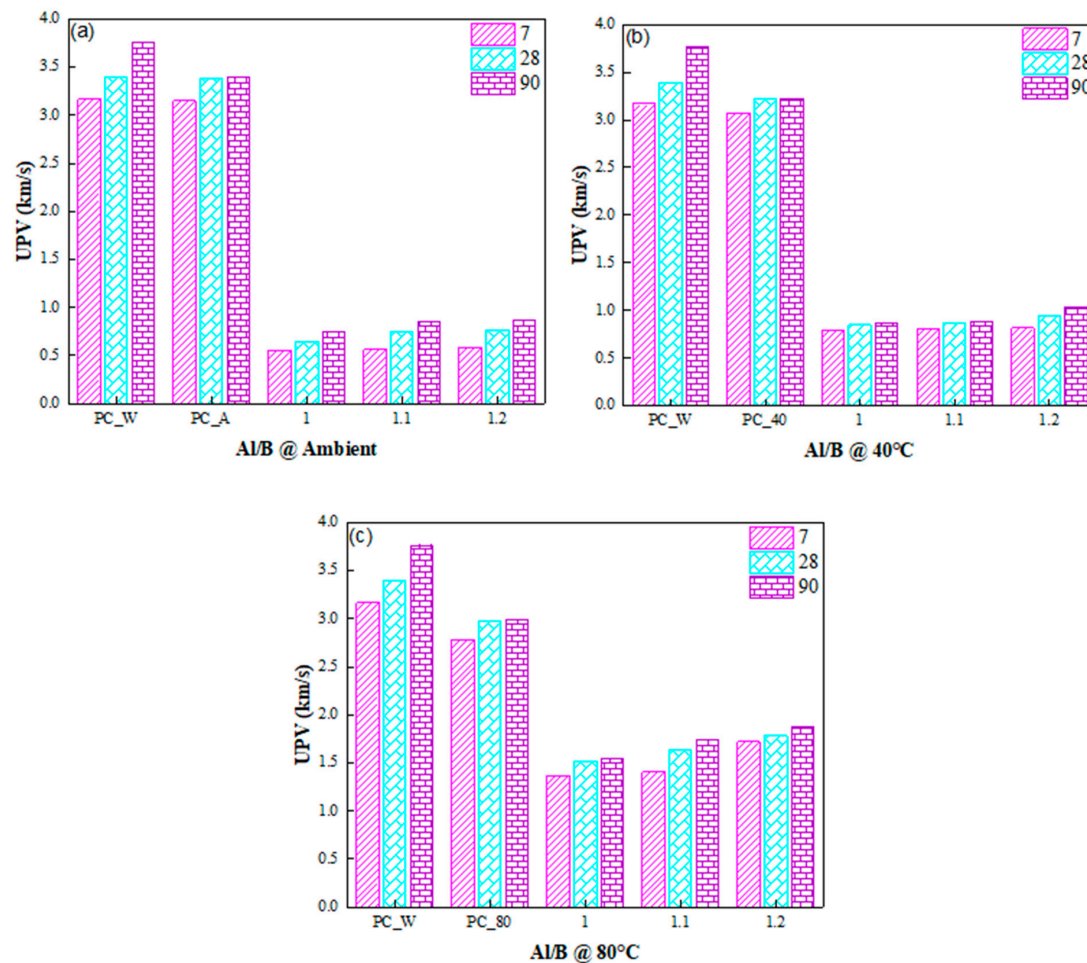


Figure 4. UPV of Mortar with Different Al/B Ratios Compared to Control Mix at Various Testing Ages and Temperatures: (a) Ambient, (b) 40 °C, and (c) 80 °C.

3.4. Flexural Strength

The flexural strength of different PAA mixes containing various Al/B ratios at different curing temperatures and ages was evaluated relative to the control mix, as shown in Figure 5. The flexural strength increased for all tested mixes as the curing age increased from 7 to 90 days. To elaborate, the flexural strength of the PAA mix with an Al/B ratio of 1.2 cured at 80 °C increased from 1.28 MPa at 7 days to 2.10 MPa at 90 days, representing an increase of 64%. This can be attributed to the densification of the mortar structure with increasing curing age [52]. The flexural strength of the PAA mixes was lower than that of the control mix. As an example, at 80 °C and 28 days of curing, the highest flexural strength recorded for the PAA mixes was 1.67 MPa; however, for control mix, the flexural strength value was 4.89 MPa. This effect was due to increased porosity, a degraded microstructure, and slower early-age strength development [53]. The flexural strength of PAA mixes was enhanced as the curing temperature increased from ambient to 80 °C. For example, for the PAA mix with an Al/B ratio of 1.2 and at 90 days of curing, the flexural strength increased from 0.56 MPa at ambient temperature to 2.10 MPa at 80 °C. This was a result of the accelerated alkali-activation reaction that facilitated the formation of binding reaction products leading to improved flexural strength at high temperatures [54]. Regarding the effect of Al/B ratio, as the ratio varied from 1 to 1.2, the flexural strength was enhanced at all curing temperatures and ages. The flexural strength at 80 °C and 90 days of curing varied from 1.16 MPa for the PAA mix with an Al/B ratio of 1.0 to 2.10 MPa for the mix with an Al/B ratio of 1.2, indicating a significant increase of approximately 81%. This was because

of the enhanced volume of alkaline activator which accelerated the dissolution, leading to a hardened alkali-activated matrix with higher flexural strength [55]. The standard deviations of the measured flexural strengths ranged from 2.65% to 5.30%, as presented in Table 4, indicating low variability among the replicate specimens.

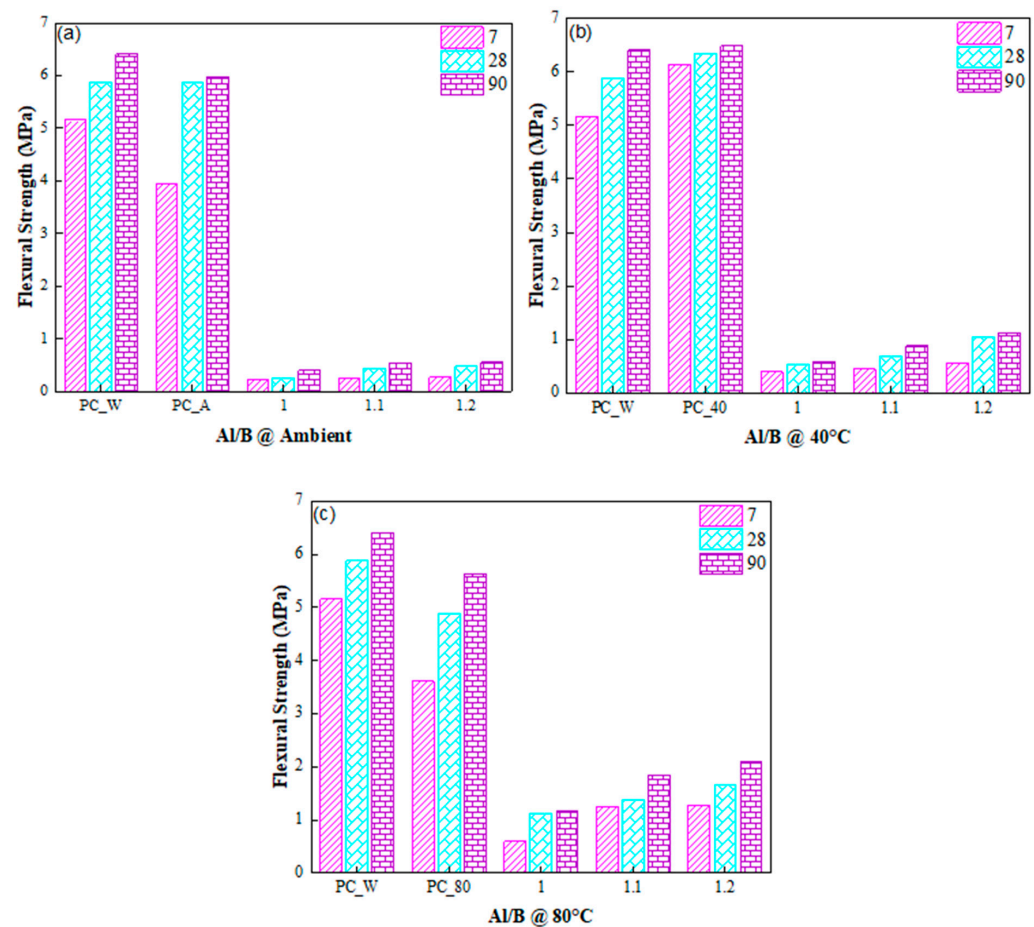


Figure 5. Flexural Strength of Mortar with Different Al/B Ratios Compared to Control Mix at Various Testing Ages and Temperatures: (a) Ambient, (b) 40 °C, and (c) 80 °C.

Table 4. Flexural Strength Standard Deviations of Mortar Mixes.

Mix ID	SD (%)		
Mix-4-A	2.65	5.30	5.30
Mix-4-40	2.65	2.65	2.65
Mix-4-80	5.30	5.30	5.30
Mix-2-A	2.65	5.30	2.65
Mix-2-40	5.30	2.65	2.65
Mix-2-80	2.65	2.65	5.30
Mix-4-A	3.71	5.30	5.30
Mix-4-40	5.30	5.30	5.30
Mix-4-80	5.30	2.65	5.30

3.5. Compressive Strength

The compressive strengths of the PAA mixes with different Al/B ratios and at various curing temperatures and ages in comparison to the control mix are presented in Figure 6. The compressive strength for all tested specimens increased with the increase in curing age from 7 to 90 days. For example, in the PAA mix with an Al/B ratio of 1.0 cured at 40 °C, compressive strength increased from 1.02 to 2.33 MPa as the curing age increased from 7 to

90 days. This was a result of the continuation and extension of the alkali-activation reaction, whereby more gel was formed, leading to higher compressive strength with time [56]. The compressive strength of the control mixes was higher than that of the PAA mixes in all tested cases. For example, the control mix after 90 days of curing and 80 °C recorded a value of 12.27 MPa, while the highest compressive strength of the PAA mixes was 4.67 MPa. This may be related to the low density of PAA leading to a weaker skeleton and the occurrence of cracks under compressive testing [57]. Comparing PAA mixes with different Al/B ratios, the compressive strength increased with the enhancement in Al/B ratio. As an example, the compressive strength for the PAA mix with an Al/B ratio equal to 1 at 80 °C and 90 days of curing was 4.44 MPa, increasing to 4.67 MPa when the Al/B ratio was 1.2, corresponding to an increase of 5.2%. This may have been due to the small amount of alkali activator which may have hindered solidification, resulting in incomplete compaction and void formation in the specimen and leading to a decrease in the compressive strength [45]. The compressive strength of the PAA mixes was enhanced as the curing temperature increased from ambient to 80 °C. For example, at 7 days of curing, in the PAA mix with Al/B equal to 1, the compressive strength increased from 0.47 MPa to 1.68 MPa when the temperature was increased from ambient to 80 °C. At high curing temperatures, dissolution is accelerated, leading to an enhanced and faster alkali-activation reaction, resulting in enhanced and higher compressive strength [58]. The standard deviations ranged from 0.44% to 6.85%, as displayed in Table 5, indicating good consistency among the specimens. The moderate compressive strength recorded in different PAA mixes allows the material to be used in different applications, including heat-cured precast non-load-bearing masonry, lightweight partition and infill elements, and internal rendering or plastering.

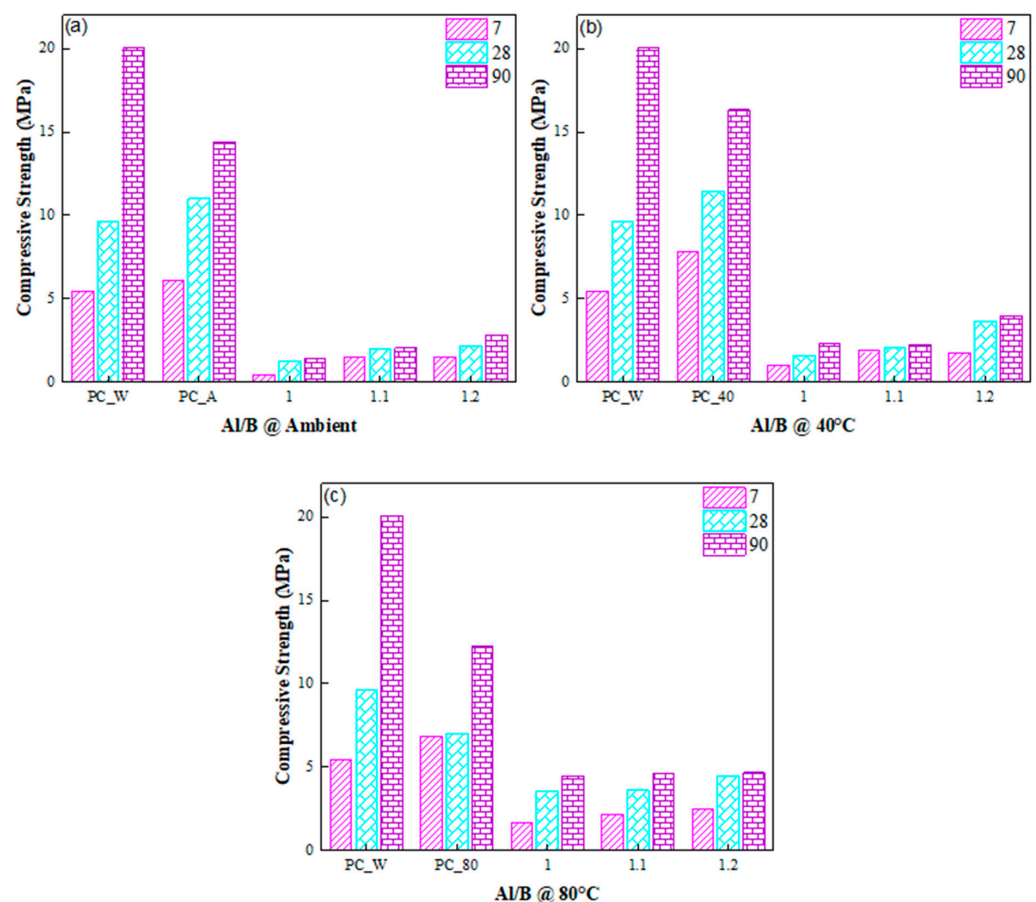


Figure 6. Compressive Strength of Mortar with Different Al/B Ratios Compared to Control Mix at Various Testing Ages and Temperatures: (a) Ambient, (b) 40 °C, and (c) 80 °C.

Table 5. Compressive Strength Test Standard Deviations of Mortar Mixes.

Mix ID		SD (%)	
Mix-4-A	4.18	0.80	5.70
Mix-4-40	6.85	3.89	1.94
Mix-4-80	1.50	2.08	0.62
Mix-2-A	3.60	2.17	3.54
Mix-2-40	2.89	2.96	4.15
Mix-2-80	4.86	2.56	2.92
Mix-5-A	5.17	2.92	2.03
Mix-5-40	4.29	2.87	5.52
Mix-5-80	6.50	0.44	0.71

3.6. Drying Shrinkage, Mass Loss and Volume Change

Figure 7 illustrates the drying shrinkage of PAA mixes with different Al/B ratios at several testing ages in comparison to the control mixes. The drying shrinkage of PAA mixes regardless of the Al/B ratio used was higher than that of the control mix. As an example, the drying shrinkage of PC_T mix at 7 days was 0.00009 mm/mm, while that for the PAA mix with Al/B equal to 1 was 0.00069 mm/mm. The alkali-activated specimens shrank more than the control mixes since less water was chemically bound during the reaction process, so a greater quantity of water remained free and able to evaporate easily during the drying shrinkage, resulting in a higher shrinkage value [59]. The drying shrinkage of all the tested mixes increased as the testing time increased from 1 day to 90 days. To elaborate, for the PAA mix with Al/B equal to 1.2, the drying shrinkage increased from 0.00324 mm/mm at day 1 to 0.01016 mm/mm at 90 days. This may have been related to the increase in water evaporation with time leading to more shrinkage [60]. Regarding the effect of Al/B ratio on drying shrinkage, as the Al/B ratio increased from 1 to 1.2, the drying shrinkage increased. At 28 days, the drying shrinkage of the PAA mixture with an Al/B ratio of 1.0 was 0.00821 mm/mm, whereas that of the mixture with an Al/B ratio of 1.2 was 0.00973 mm/mm. The high Al/B ratio may have been left unreacted due to the large amount of liquid, leading to more free water evaporation and resulting in higher shrinkage [61]. Figure 8 shows the percentage of mass loss and volume change for different mortar mixes containing different Al/B ratios in comparison to the control mix. Both the volume and mass follow the same trend as drying shrinkage, with the mass and volume loss increasing due to the evaporation of water [62].

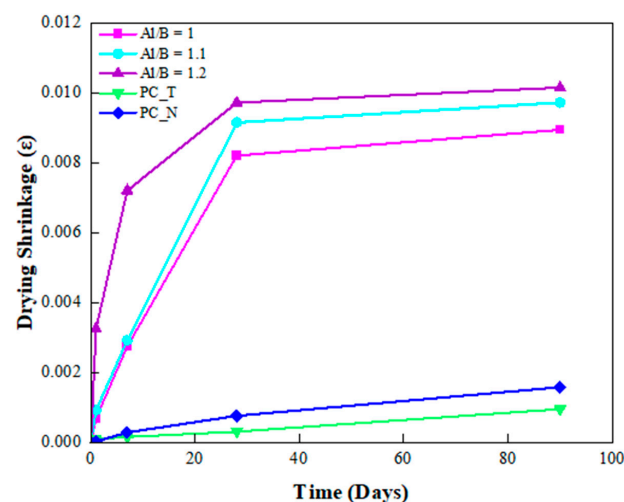


Figure 7. Drying Shrinkage of Mortar with Different Al/B Ratios Compared to Control Mix at Various Testing Ages.

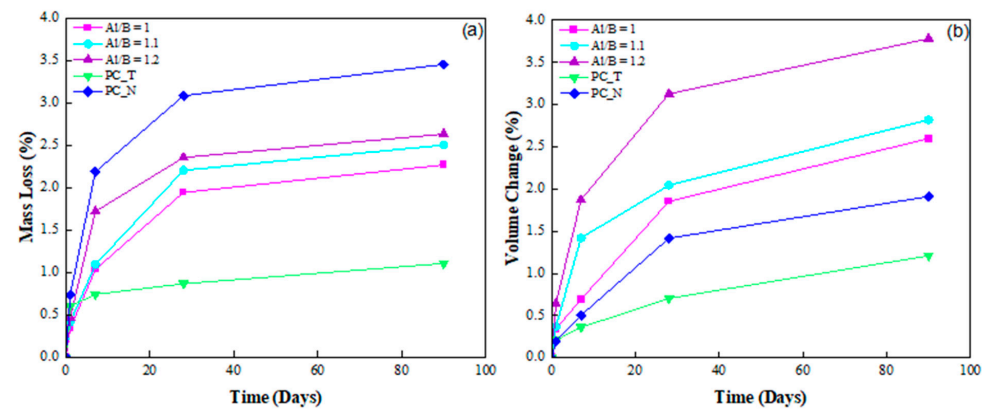


Figure 8. Mass Loss and Volume Change of Mortar Due to Drying Shrinkage with Different Al/B Ratios Compared to Control Mix at Various Testing Ages: (a) Mass Loss and (b) Volume Change.

3.7. Autogenous Shrinkage, Mass Loss, and Volume Change

The variation of autogenous shrinkage as a function of the Al/B ratio at several testing ages in comparison to the control mix is presented in Figure 9. The results reveal that the autogenous shrinkage of PAA mixes was higher than that of the PC mixes regardless of the Al/B ratio used and the testing day studied. To clarify, at 90 days, the PC_T mix had a value of 0.00098 mm/mm, while the lowest value recorded for the PAA mix with an Al/B ratio equal to 1 was 0.00387 mm/mm. This was due to the formation of micro-cracks from the contracting volume of the specimen, resulting in higher shrinkage [11]. Autogenous shrinkage increased with testing age [63]. For example, the autogenous shrinkage of PAA mix with an Al/B ratio equal to 1.2 varied from 0.00092 mm/mm to 0.00529 mm/mm as the time increased from 1 day to 90 days. The autogenous shrinkage values were smaller than the corresponding drying-shrinkage values. The autogenous shrinkage of PAA mixes increased as the Al/B ratio increased from 1 to 1.2. To clarify, at 28 days, the autogenous shrinkage increased from 0.00270 mm/mm to 0.00346 mm/mm when the Al/B ratio changed from 1 to 1.2. When the liquid content is too low or too high relative to the binder content, internal tensile stresses can occur, leading to an increase in the shrinkage values and resulting in micro-cracks [61]. Figure 10 displays the mass loss and volume change for the different mortar mixes due to autogenous shrinkage; all specimens' recorded values were below 2.5%.

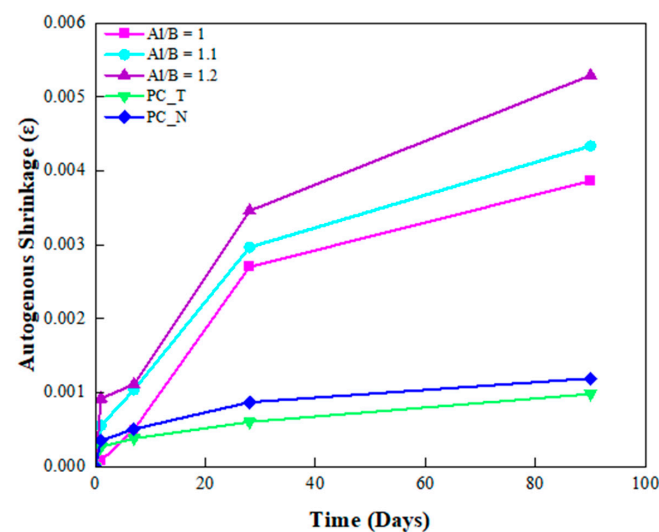


Figure 9. Autogenous Shrinkage of Mortar with Different Al/B Ratios Compared to Control Mix at Various Testing Ages.

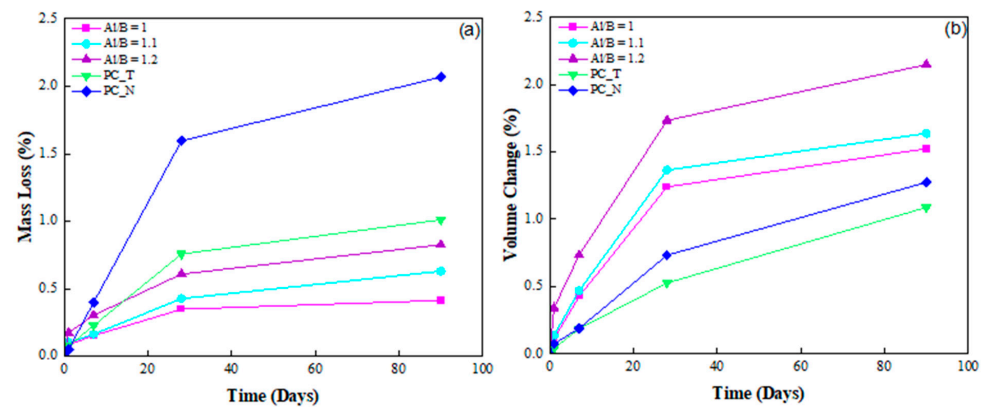


Figure 10. Mass Loss and Volume Change of Mortar Due to Autogenous Shrinkage with Different Al/B Ratios Compared to Control Mix at Various Testing Ages: (a) Mass Loss and (b) Volume Change.

3.8. Chemical Shrinkage

Figure 11 shows the chemical shrinkage of different PAA mixes with various Al/B ratio in comparison to control mix at several testing days. Comparing the PC mix with PAA mixes, the PC mix had chemical shrinkage values higher than those for the PAA mixes. To illustrate, the chemical shrinkage of PC mix at 28 days had a value of 0.125 mL/g while the highest value of chemical shrinkage recorded for any of the PAA mixes was 0.080 mL/g for the mix with Al/B equal to 1.2. This is because of the difference between the reaction mechanisms of alkali-activated and conventional cement-based mortars; the former undergoes an alkali-activation process, whereas the latter undergoes hydration and consumes water, resulting in higher chemical-shrinkage values [64]. For all the tested mixes, chemical shrinkage increased with time. For example, the value for the PAA mixture with an Al/B ratio of 1.0 changed from 0.007 to 0.147 mL/g as age increased from 1 to 90 days. Concerning the effect of Al/B ratios on chemical shrinkage, similarly to the drying and autogenous shrinkage, the chemical shrinkage increased with the increase in Al/B ratio [65]. This may have been related to the pore formation and the contraction that occurred with higher Al/B [61].

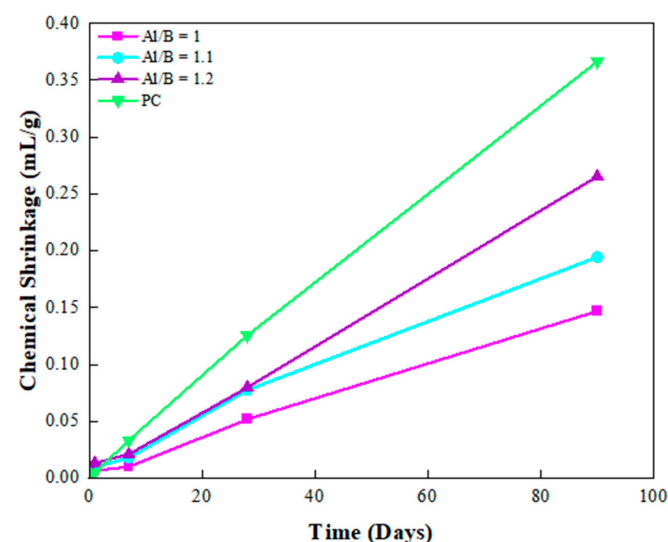


Figure 11. Chemical Shrinkage of Mortar with Different Al/B Ratios Compared to Control Mix at Various Testing Ages.

4. Correlations Between Different Properties

All regression coefficients/equations developed in this subsection are dataset-specific due to the grouping together of PC and PAA mortar data for the analyses. Also, in

case of analyses per mix, the results obtained are applicable for these mixes within these ranges only. Therefore, all data are applicable only over the ranges of density, UPV, strength, and shrinkage examined in this study and should not be considered universal predictive models.

4.1. Correlation Between Compressive Strength and Flexural Strength with Density

Figure 12 displays the correlation between the density and strength for mortar mixes including PAA and control specimens at all curing temperatures and ages. Figure 12a shows the correlation between compressive/flexural strength versus density with respect to curing ages, while Figure 12b displays the correlation between compressive strength and density with respect to each mix on all curing ages. A good linear relationship between the two tested properties is revealed, where an increase in compressive/flexural strength is associated with an increase in the density. The correlation coefficients show values above 0.87 in Figure 12a and above 0.65 in Figure 12b. Figure 13 indicates that L increased from 0.006 at 7 days to 0.016 at 90 days for compressive strength (CS) and from 0.005 to 0.006 for flexural strength (FS) as the time increased from 7 to 90 days. In this dataset, the relationship between density and strength became steeper with age. K became more negative with age and should be interpreted as a regression parameter, not a strength value with physical meaning.

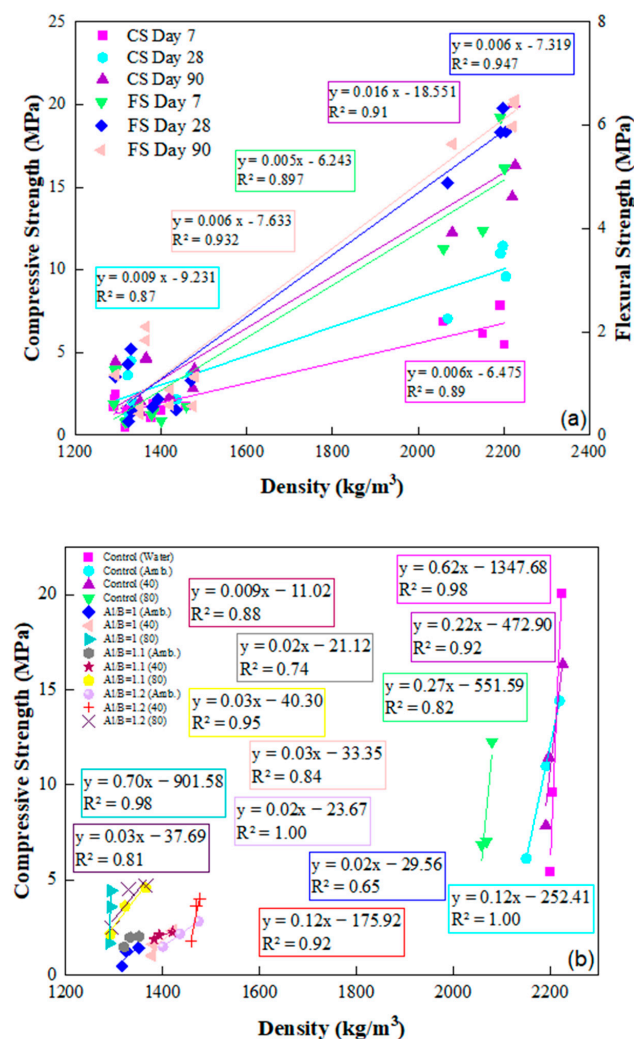


Figure 12. Correlation Between Strength and Density of Different Mortar Mixes Including Control and PAA Mixes With Various Al/B Ratios: (a) Compressive/Flexural Strength vs. Density as Function of Day; (b) Compressive Strength vs. Density as Function of Mix.

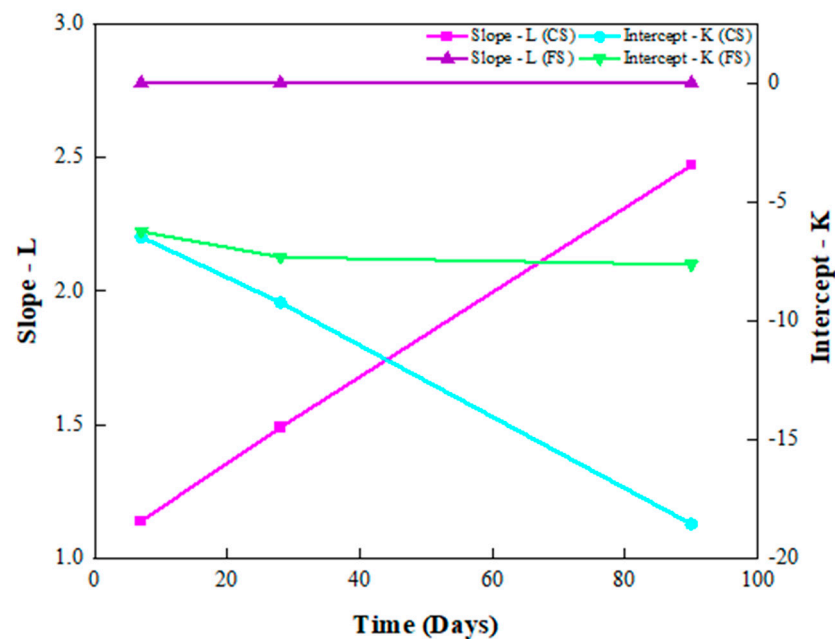


Figure 13. Slope (L) and Intercept (K) for the Linear Relationship Between Compressive/Flexural Strength and Density at Different Testing Ages.

4.2. Correlation Between Compressive Strength and UPV

Figure 14 shows the exponential relationship between UPV and compressive strength, which can be considered a good relationship since the correlation coefficients recorded for all tested mixes are above 0.83. As the compressive strength of tested mixes increased, the UPV also increased.

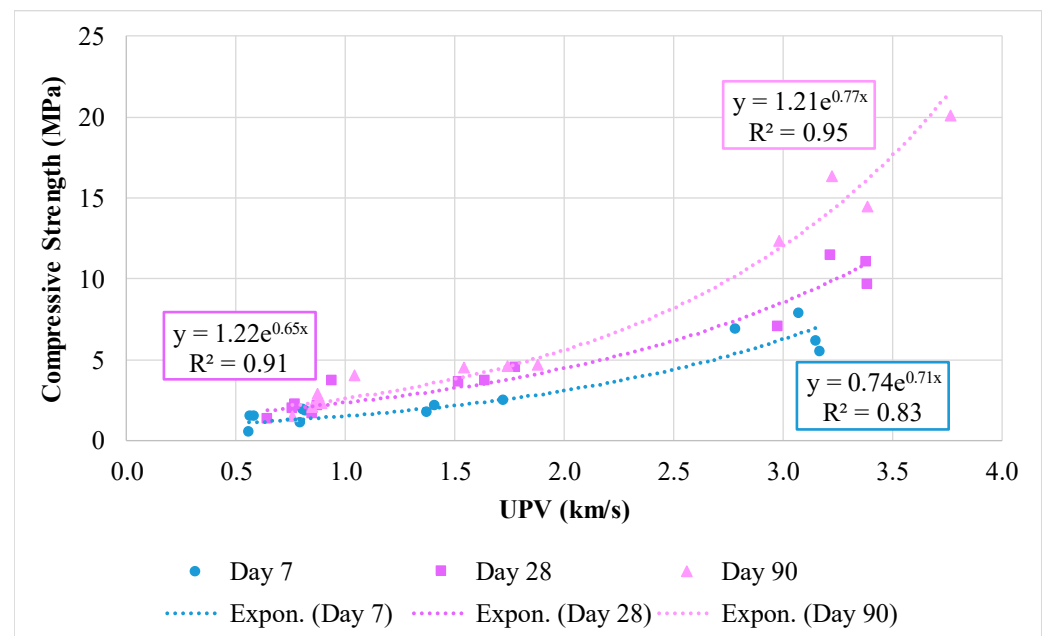


Figure 14. Correlation Between Compressive Strength and UPV of Different Mortar Mixes Including Control and PAA Mixes with Various Al/B Ratios.

4.3. Correlation Between Compressive Strength and Flexural Strength

A linear relationship between flexural and compressive strength was present for different PAA and control mixes at different curing ages, as presented in Figure 15. Figure 15a displays the correlation with respect to different curing ages and Figure 15b presents the

correlations between compressive and flexural strength per mix. The relationship can be considered good, with all correlation coefficients showing values above 0.91 in Figure 15a and above 0.51 in Figure 15b. The flexural strength and compressive strength are proportional, whereby an increase in one property leads to an increase in the other. Figure 16 indicates that the slope L increased from 1.14 at 7 days to 2.47 at 90 days. The slope indicates that the compressive strength increased at a faster rate with respect to the flexural strength as the curing age increased. This trend of age-dependency on slope L means that a single conversion equation between compressive and flexural strength cannot characterize all curing ages.

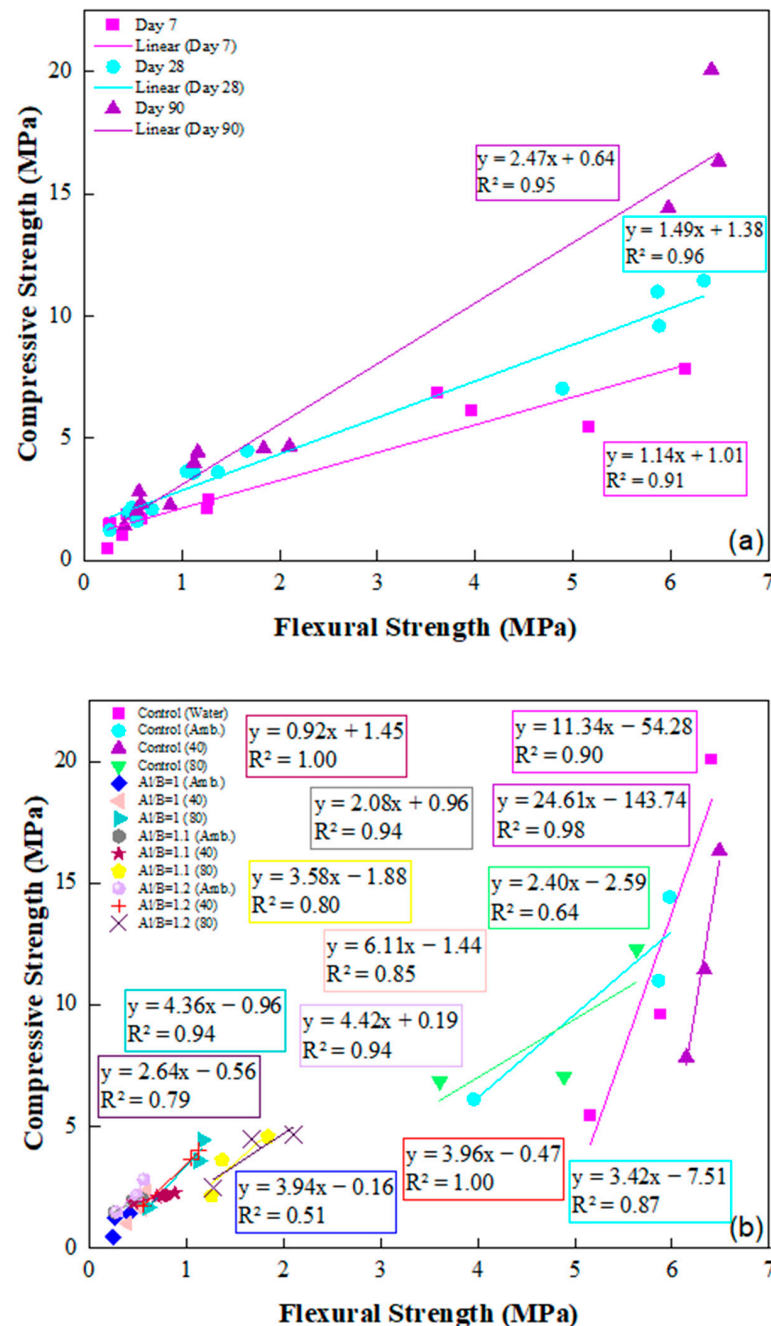


Figure 15. Correlation Between Compressive Strength and Flexural Strength of Different Mortar Mixes Including Control and PAA Mixes with Various Al/B Ratios: (a) Compressive Strength vs. Flexural Strength as Function of Day; (b) Compressive Strength vs. Flexural Strength as Function of Mix.

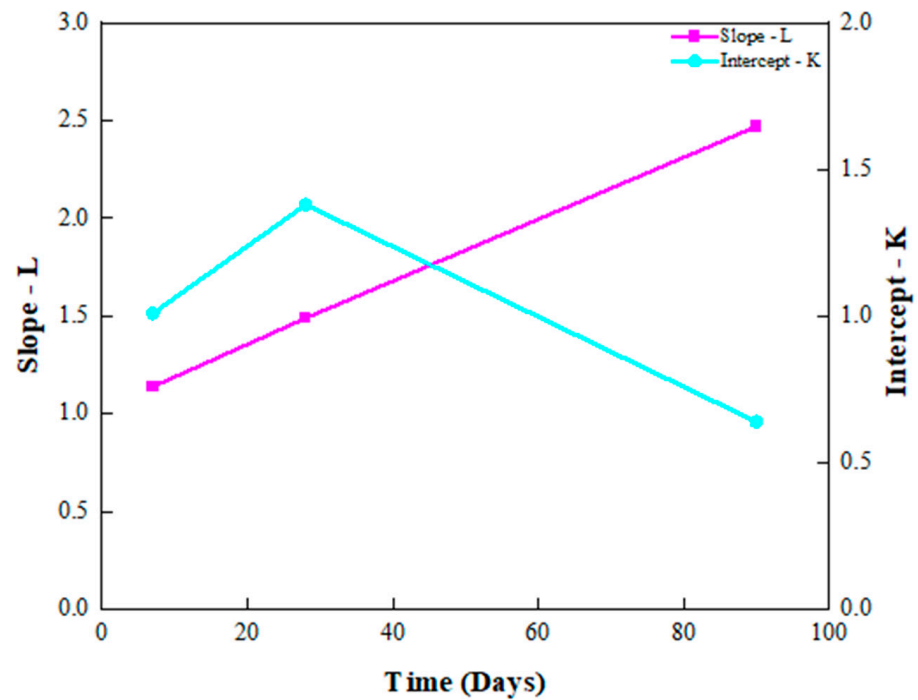


Figure 16. Slope (L) and Intercept (K) for the Linear Relationship Between Compressive Strength and Flexural Strength at Different Testing Ages.

4.4. Correlation Between Autogenous Shrinkage and Drying Shrinkage

Figure 17 plots a good linear relationship between drying and autogenous shrinkage with respect to different A1/B ratios at several curing ages. As drying shrinkage increases, autogenous shrinkage becomes higher. The correlation coefficients exhibit a minimum value of 0.71.

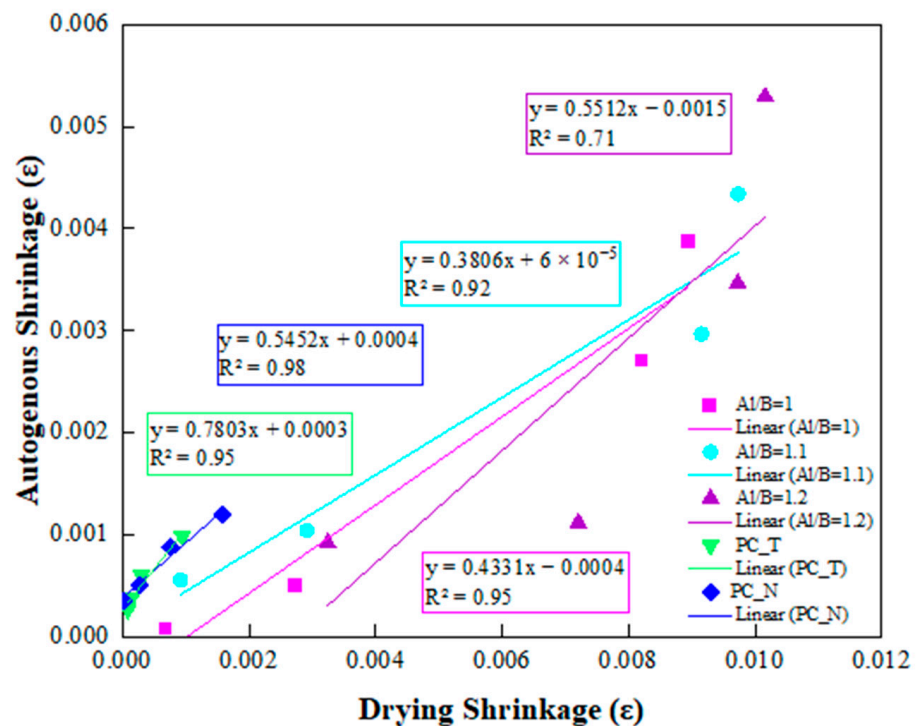


Figure 17. Correlation Between Autogenous Shrinkage and Drying Shrinkage of Different Mortar Mixes Including Control and PAA Mixes with Various A1/B Ratios.

4.5. Correlation Between Drying Shrinkage, Mass Loss and Volume Change

Figure 18 presents the linear correlations between mass loss and volume change with drying shrinkage for several PAA and control mixes. There appears to be a good relationship between the studied properties where the minimum correlation coefficients for mass loss and volume change are 0.77 and 0.87, respectively. Both mass loss and volume change follow the same trend as drying shrinkage.

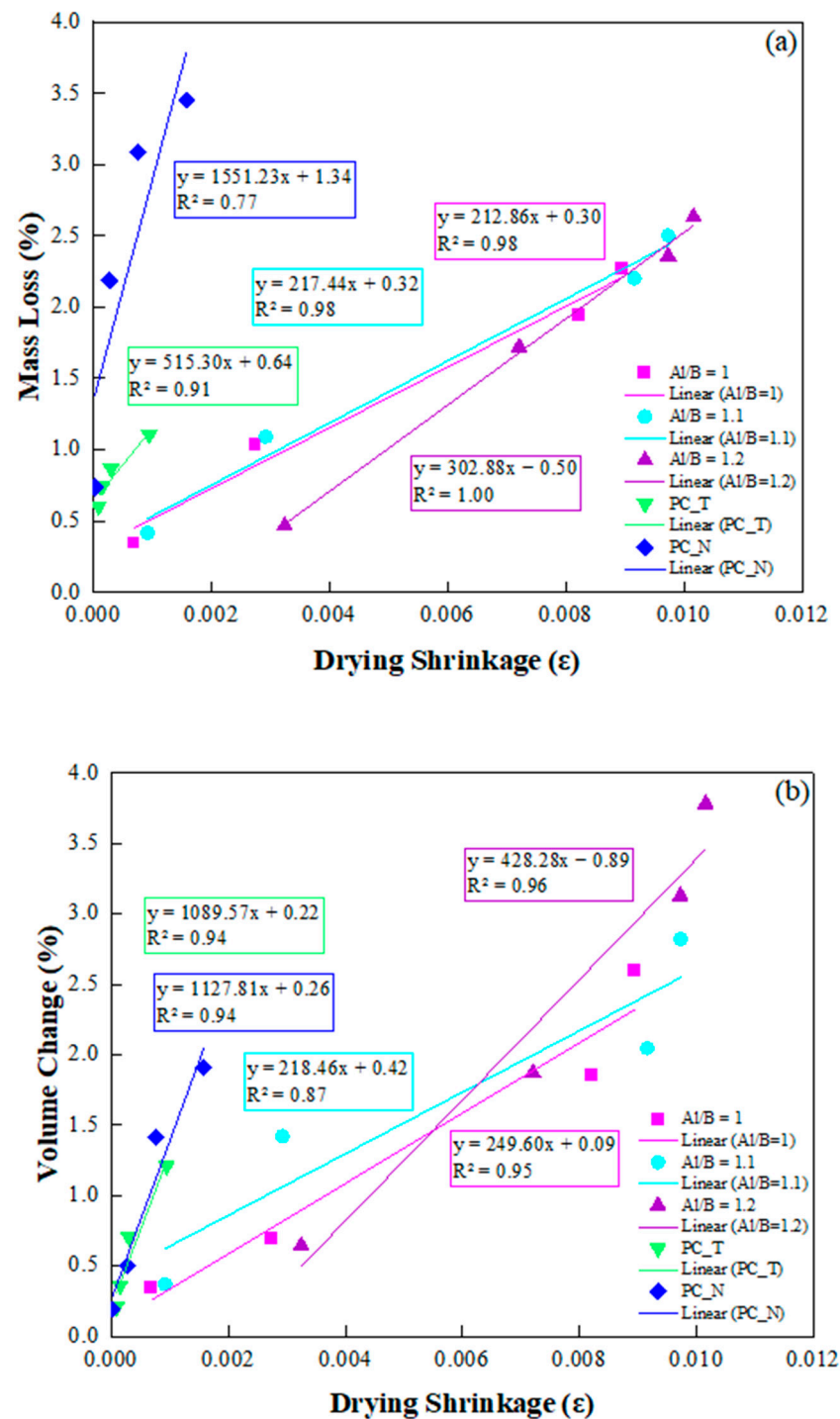


Figure 18. Correlation Between Mass Loss, Volume Change and Drying Shrinkage of Different Mortar Mixes Including Control and PAA Mixes with Various Al/B Ratios: (a) Mass Loss and (b) Volume Change.

4.6. Correlation Between Autogenous Shrinkage, Mass Loss and Volume Change

Figure 19 plots the relationships between mass loss and volume change with autogenous shrinkage. There appears to be a good correlation, with the correlation coefficients showing a minimum value of 0.93 and 0.95 for mass loss and volume change, respectively. Both properties follow a similar trend with autogenous shrinkage.

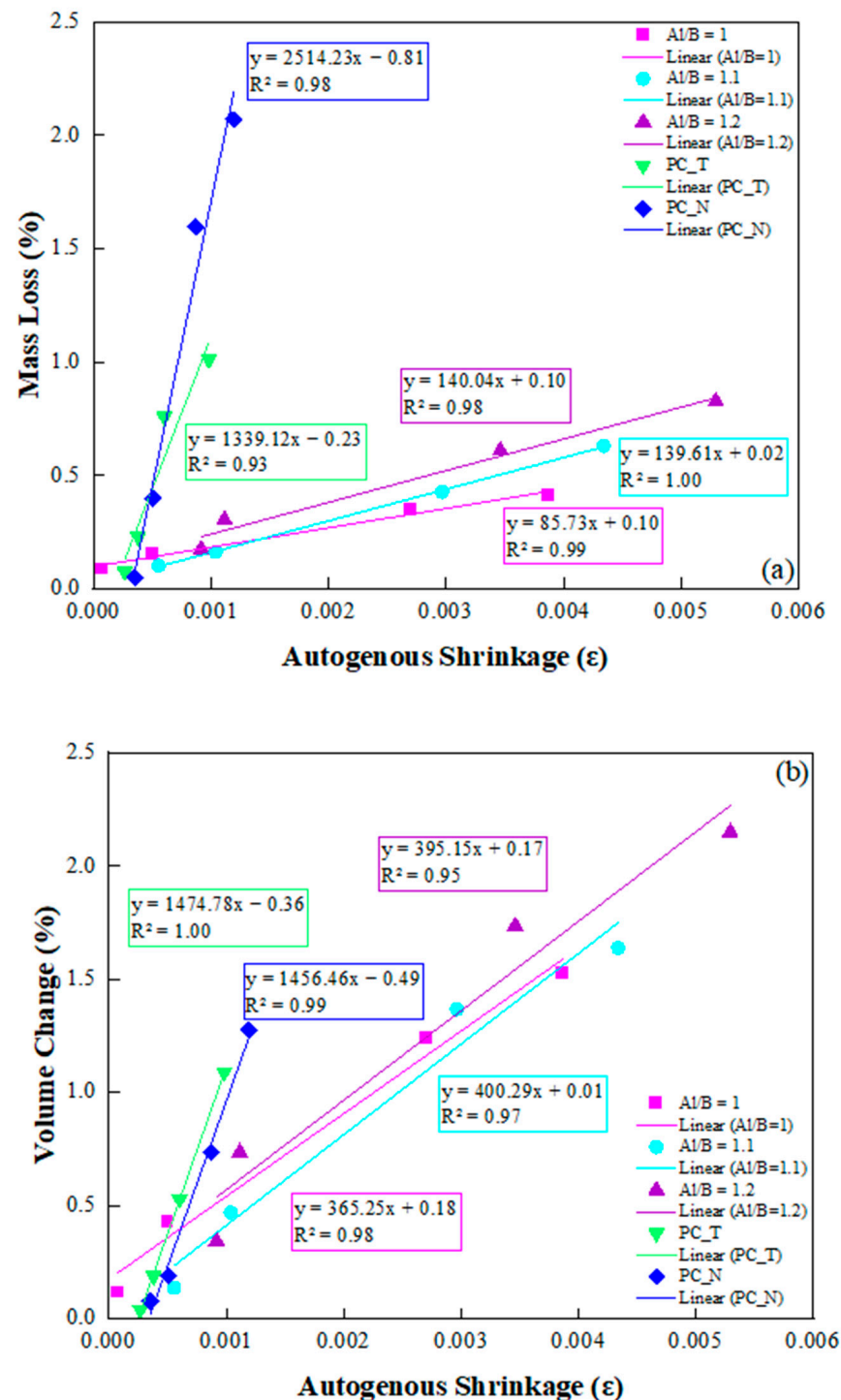


Figure 19. Correlation Between Mass Loss, Volume Change and Autogenous Shrinkage of Different Mortar Mixes Including Control and PAA Mixes with Various Al/B Ratios: (a) Mass Loss and (b) Volume Change.

5. ANOVA and Tukey Analysis of the Impact of Al/B Ratio and Temperature on PAA Mixes

To evaluate the quantitative data differences between several Al/B ratios and various temperatures, a one-way analysis of variance (ANOVA) was performed on the dimensional stability test results and a two-way analysis of variance (ANOVA) was conducted for the mechanical properties. Both analyses were evaluated at a 95% confidence level ($\alpha = 0.05$). All properties including density, UPV, flexural strength, compressive strength, drying, and autogenous shrinkage were examined at 7, 28 and 90 days of curing.

Two hypotheses were used in the ANOVA study, as follows:

1. The null hypothesis (H_0) states that there is no statistically significant difference when the p -value is greater than 0.05;
2. The alternative hypothesis (H_1) states that there is a statistically significant difference when the p -value is below 0.05, indicating that at least one mixture differs.

The Tukey HSD post hoc test was used to group the mixes. In the reported results, the same letter is assigned to mixtures that do not differ significantly, whereas different letters are assigned to mixtures that differ significantly. This method allows the identification of the best Al/B used in PAA mixes and the best temperature for curing purposes.

Table 6 presents the p -values for different tested parameters with factor Al/B at 7, 28, and 90 days of curing. The results show that the Al/B ratio does not have a significant effect on all parameters tested at 28 days of curing; density, UPV, drying shrinkage, and autogenous shrinkage do not differ significantly with Al/B ratio. However, the Al/B ratio has a statistically significant effect on flexural and compressive strengths, for which the p -values are below 0.05. At 7 and 90 days, the trend is similar for that at 28 days, except for UPV at 90 days and for drying shrinkage at 7 days, where a statistically significant effect is recorded.

Table 6. ANOVA p -Values for the Tested Parameters with Factor Al/B.

Curing Age (Days)	Parameter	Density	UPV	Flexural Strength	Compressive Strength	Drying Shrinkage	Autogenous Shrinkage
7	p -value	0.078	0.017	1.34×10^{-6}	3.49×10^{-5}	3.97×10^{-4}	0.308
28		0.124	0.056	1.43×10^{-7}	4.87×10^{-14}	0.478	0.288
90		0.037	1.70×10^{-5}	2.32×10^{-8}	1.88×10^{-5}	0.536	0.031

Table 7 shows the p -values for different tests with the temperature factor at 7, 28, and 90 days of curing. Results reveal that the temperature has an effect on all parameters except density at 28 days, for which the p -value recorded is above 0.05. So, the Al/B ratio affects the strength of alkali-activated mortar mixes, with an increase in the ratio leading to a stronger matrix. High temperature affects the strength and quality of the PAA mixes, whereby increasing the temperature results in a better matrix with fewer voids and stronger structure.

Table 7. ANOVA p -Values for the Tested Parameters with Factor Temperature.

Curing Age (Days)	Parameter	Density	UPV	Flexural Strength	Compressive Strength
7	p -value	0.00369	4.75×10^{-9}	3.25×10^{-10}	3.05×10^{-5}
28		0.064	8.23×10^{-8}	7.38×10^{-11}	6.02×10^{-16}
90		0.020	2.22×10^{-11}	2.01×10^{-11}	2.24×10^{-8}

Table 8 displays the *p*-values for different tests with an interaction between the two factors. For example, at 28 days of curing, only flexural and compressive strength record *p*-values below 0.05, indicating a statistically significant effect.

Table 9 displays the grouping of mixes for the tested parameters for the Al/B and temperature factors, while Table 10 presents the grouping of mixes for the tested parameters for the interaction between Al/B and temperature factors. With regard to density, the Al/B has no effect on the grouping of the mixes except at 90 days; however, the temperature affects the grouping of the mixes except at 28 days. The best mix is that with an Al/B ratio of 1.2 at a curing temperature of 40 °C. Regarding UPV, the best mix has an Al/B ratio of 1.2 and was cured at 80 °C, and the worst mix is the mix with Al/B equal to 1 and cured at ambient temperature (20 °C). For flexural and compressive strength, the best mix is the PAA mortar with Al/B equal to 1.2 and cured at 80 °C. Regarding the dimensional-stability parameters, including drying and autogenous shrinkage, there is no significant effect of either Al/B or temperature except for drying shrinkage at 7 days. So, the grouping is the same for all the PAA mixes.

Table 8. ANOVA *p*-Values for the Tested Parameters with Interaction Between Two Factors Including Al/B and Temperature.

Curing Age (Days)	Parameter	Density	UPV	Flexural Strength	Compressive Strength
7	<i>p</i> -value	0.58885	0.024	4.73×10^{-6}	0.27389
28		0.8431	0.70961	0.00166	9.00×10^{-11}
90		0.47921	0.00986	1.01×10^{-5}	0.00187

Table 9. Tukey HSD Grouping of PAA Mortar Mixes for Al/B and Temperature Factors.

Curing Age (Days)	Factor	Density		UPV		Flexural Strength		Compressive Strength		Drying Shrinkage		Autogenous Shrinkage		
7	Factor: Al/B													
		Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	
	1.2	1384.77	A	1.040	A	0.706	A	1.903	A	0.00721	B	0.00111	A	
	1.1	1331.90	A	0.931		0.656	A	1.828	A	0.00292		0.00104	A	
	1	1327.99	A	0.908		0.413	B	1.054	B	0.00274	A	0.0005	A	
	Factor: Temperature													
	80	1292.32		B	1.504	A	1.044	A	2.095	A				
	40	1406.45	A		0.807		0.469	B	1.548		B			
	20	1345.90	A	B	0.569		0.263	C	1.143		C			
	28	Factor: Al/B												
		Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	
1.2		1411.91	A	1.164	A	1.069	A	3.438	A	0.00973	A	0.00346	A	
1.1		1349.54	A	1.093	A	0.838	B	2.576	B	0.00916	A	0.00296	A	
1		1332.81	A	1.010	A	0.644	C	2.149	C	0.00821	A	0.00270	A	
Factor: Temperature														
80		1315.04	A		1.650	A	1.388	A	3.900	A				
40		1414.45	A		0.892	B	0.763	B	2.454	B				
20		1364.78	A		0.725		0.400	C	1.809	C				
90		Factor: Al/B												
		Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	Mean	Groups	
	1.2	1439.13	A	1.266	A	1.263	A	3.827	A	0.01016	A	0.00529	A	
	1.1	1379.10	A	B	1.160	B	1.088	B	2.962	B	0.00973	A	0.00434	A
	1	1354.95		B	1.058	C	0.719	C	2.731	B	0.00895	A	0.00387	B
	Factor: Temperature													
	80	1341.21		B	1.72	A	1.700	A	4.566	A				
	40	1439.84	A		0.934	B	0.863	B	2.863	B				
	20	1392.12	A	B	0.826		0.506	C	2.091	C				

Table 10. Tukey HSD Grouping of PAA Mortar Mixes for the Interaction Between Al/B and Temperature Factors.

Curing Age (Days)	Density				UPV				Flexural Strength				Compressive Strength			
	Al/B	Temperature	Mean	Groups	Al/B	Temperature	Mean	Groups	Al/B	Temperature	Mean	Groups	Al/B	Temperature	Mean	Groups
7	1.2	40	1458.98	A	1.2	80	1.72	A	1.2	80	1.28	A	1.2	80	2.47	A
	1.2	20	1400.39	A	1.1	80	1.42	B	1.1	80	1.26	A	1.1	80	2.14	A
	1.1	40	1382.62	A	1	80	1.37	B	1	80	0.6	B	1.1	40	1.87	A
	1	40	1377.73	A	1.2	40	0.82	C	1.2	40	0.56	B	1.2	40	1.76	A
	1.1	20	1320.90	A	1.1	40	0.81	C	1.1	40	0.45	B C	1	80	1.68	A
	1	20	1316.41	A	1	40	0.80	C	1	40	0.39	C	1.2	20	1.49	A
	1.2	80	1294.92	A	1.2	20	0.58	C	1.2	20	0.28	D	1.1	20	1.48	A
	1.1	80	1292.19	A	1.1	20	0.57	C	1.1	20	0.26	D	1	40	1.02	A
	1	80	1289.84	A	1	20	0.56	C	1	20	0.24	D	1	20	0.47	A
28	1.2	40	1470.12	A	1.2	80	1.78	A	1.2	80	1.67	A	1.2	80	4.49	A
	1.2	20	1435.94	A	1.1	80	1.64	A	1.1	80	1.37	B	1.2	40	3.66	B
	1.1	40	1392.77	A	1	80	1.53	A	1	80	1.13	C	1.1	80	3.62	B
	1	40	1380.47	A	1.2	40	0.94	A	1.2	40	1.05	C	1	80	3.59	B
	1.1	20	1333.40	A	1.1	40	0.88	A	1.1	40	0.69	D	1.2	20	2.17	C
	1.2	80	1329.69	A	1	40	0.85	A	1	40	0.54	D E	1.1	40	2.10	C D
	1	20	1325.00	A	1.2	20	0.77	A	1.2	20	0.49	E	1.1	20	2.01	D
	1.1	80	1322.46	A	1.1	20	0.76	A	1.1	20	0.45	E	1	40	1.61	E
	1	80	1292.97	A	1	20	0.65	A	1	20	0.26	F	1	20	1.25	F
90	1.2	40	1477.9	A	1.2	80	1.88	A	1.2	80	2.1	A	1.2	80	4.67	A
	1.2	20	1474.2	A	1.1	80	1.74	A	1.1	80	1.84	B	1.1	80	4.59	A
	1.1	40	1421.1	A	1	80	1.54	B	1	80	1.16	C	1	80	4.44	A
	1	40	1420.5	A	1.2	40	1.04	C	1.2	40	1.13	C	1.2	40	4.00	A
	1.2	80	1365.2	A	1.1	40	0.89	D	1.1	40	0.88	D	1.2	20	2.81	B
	1.1	80	1364.8	A	1.2	20	0.87	D	1	40	0.58	E	1	40	2.33	B
	1.1	20	1351.4	A	1	40	0.87	D	1.2	20	0.56	E	1.1	40	2.26	B
	1	20	1350.8	A	1.1	20	0.85	D	1.1	20	0.54	E	1.1	20	2.03	B
	1	80	1293.6	A	1	20	0.76	D	1	20	0.41	E	1	20	1.42	C

6. Conclusions

This study demonstrates that *Phragmites australis* ash can be used as the sole precursor in low-density alkali-activated mortar; however, its performance is governed by a trade-off among activator content, heat curing, mechanical capacity, and dimensional stability. The principal conclusions are as follows:

- Raising the Al/B ratio from 1.0 to 1.2 resulted in an increased workability and mechanical properties. An average increase in flexural strength from 0.64 MPa to 1.07 MPa and a compressive strength of 2.15 MPa to 3.44 MPa was observed at 28 days. All the increases were statistically significant except for density, UPV, and both types of shrinkage which showed no significance at 28 days. This would indicate that a ratio of 1.2 is ideal to improve the strength properties of the mortar within the ranges tested;
- Temperature had a greater impact on the development of mechanical properties than the Al/B ratio. An increase in curing temperature from 20 to 80 °C yielded an average increase in UPV value from 0.72 to 1.65 km/s, flexural strength from 0.40 MPa to 1.38 MPa, and compressive strength from 1.81 to 3.90 MPa at 28 days. The increase in temperature had no significant effect on density at 28 days. It is clear that heat curing can be used to improve the mechanical properties of the PAA mortar. Poor strength development at room-temperature curing is still a drawback;
- It can also be concluded from the interactions that the Al/B ratio should not be taken out of context with respect to temperature. There was a significant interaction effect between the Al/B ratio and temperature on flexural strength for all ages and on compressive strength at 28 days and 90 days. Care should therefore be taken when determining how much activator to add to the mix as well as what temperature to cure it at;
- The PAA mortar that had the highest 90-day flexural and compressive strength values was the mix with an Al/B ratio of 1.2 that was cured at 80 °C, with values of 2.10 MPa and 4.67 MPa respectively. Although an increase was seen, it still fell short of the Portland-cement control mix by about 62%. This means that although we were able to improve the strength properties of the mortar, it still could not compete with conventional Portland-cement mortar;
- It was shown that all the PAA mixes had a density of less than 1500 kg/m³. However, they showed higher values for drying and autogenous shrinkage compared to the control mix. Despite having a lower chemical shrinkage, an increase in shrinkage was observed as the Al/B ratio increased, but this was not significant at 28 days. Shrinkage should be examined further before this mortar can be used in practice;
- PAA mortar could be used as a low-density mortar to cast precast non-load bearing masonry units. Other uses include light-weight partition walls and render/plaster for interior applications. More tests need to be conducted in order to check the durability of this mortar, and the shrinkage values obtained from the present experiments need to be tested using standard methods. Further research should also look at the microstructure of the mortar and testing at a unit scale.

7. Limitations and Recommendations for Future Work

These findings are limited to one PAA processing method, a relatively narrow Al/B range, and a single activator formulation consisting of 12 M sodium hydroxide and an SS/SH ratio of 2.5. Moreover, the reaction products and pore structure were not directly characterized, while long-term durability and component-level performance are outside the study scope. Future research will examine broader Al/B ratios and sodium-hydroxide concentrations, evaluate limited Portland-cement addition where improved ambient-cured strength is required, and incorporate microstructural characterization. Water absorption,

sorptivity, expansion, acid resistance, bond strength, thermal and fire performance, and standardized shrinkage measurements should also be investigated before practical applications are proposed.

Author Contributions: Conceptualization, L.W.E., H.G. and J.K.; methodology, L.W.E., A.E. and H.G.; software, L.W.E.; validation, L.W.E., J.K. and A.E.; formal analysis, L.W.E. and H.A.; investigation, L.W.E.; resources, L.W.E. and J.K.; data curation, L.W.E.; writing—original draft preparation, L.W.E.; writing—review and editing, A.E., J.K., H.A. and H.G.; visualization, A.E., J.K., H.A. and H.G.; supervision, A.E., J.K. and H.G.; project administration, A.E., J.K. and H.G. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Data are contained within the article.

Acknowledgments: The authors would like to acknowledge the help of the technicians at the materials laboratory at BAU.

Conflicts of Interest: The authors declare no conflicts of interest.

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