

Article

An Experimental Study of the Effects of Low-Calcium Fly Ash on Type II Concrete

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Abstract: In this study, the compressive strength and the permeation properties of fly ash-based Geopolymer were experimentally investigated. Type 2 Portland cement (T2PC) was partially or entirely replaced with 0, 10, 20, 30, 50, 70, and 100% of fly ash (FA). The laboratory tests were conducted for compressive strength at 7, 28, and 90 days, and permeation properties such as water absorption at 7 and 28 days. The main goal was to produce eco-friendly concrete with high strength and low permeability through blending cementitious materials including low Calcium (Ca) (T2PC and FA) for protecting concrete against sulphate attacks and other chemically destructive compounds in the environment. This study focused on the effectiveness of the curing period, combinations of chemical activators by varying the molarity of alkaline solutions between 4.16 and 12.96 M and keeping the sodium silicate (SS) to sodium hydroxide (SH) by the weight ratio of 2.5. Lab observations from this study demonstrated that the compressive strength was enhanced with the increment in fly ash content at all ages, with optimum being at 20% as the replacement of T2PC.

Keywords: fly ash; geopolymer concrete; Type II Portland cement; compressive strength



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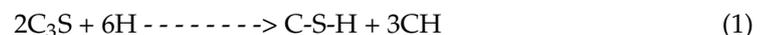
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1. Introduction

Geopolymeric admixtures such as artificial pozzolans including fly ash (FA), silica fume (SF), and natural pozzolans including volcanic ash (VA) and opaline shade chert (OSC) as well as calcined diatomaceous earth (CDE) are partially used as the replacement of ordinary Portland cement (OPC) to improve the strength and durability of concrete so that these silicon or silicon-alumina materials containing activated silica and secondary cementitious properties do not exclusively have adhesive feature in the water, but that they react with calcium hydroxide released from the hydration process of Portland cement and H₂O (Equations (1) and (2)), resulting in the formation of calcium silicates (Equation (3)) with the adhesive properties, which is similar to the reaction of cement silicates and H₂O [1–10]. It is well known that the chemical reaction in the Portland hydration can be written as



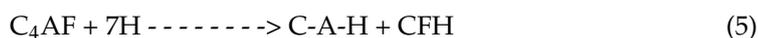
whereas C₃S is 3CaO·SiO₂ (three silicate calcium), C₂S is 2CaO·SiO₂ (dicalcium silicate), and SH basically is pozzolanic substances including Si(OH)₄ or H₄SiO₄.

The reaction formulas clearly show that the calcium silicate hydrate (C-S-H) and calcium hydroxide (C-H) are the two main products of the hydration for Portland cement in which the C-S-H chemical chain is C₃S₂H₃ comprised of C, S, and H, representing CaO, SiO₂, and H₂O, respectively. The C-H combination is also calcium hydroxide, Ca(OH)₂. (C-S-H) is a high-strength compound so that the compressive strength of hardened concrete is attributed to the formation of C-S-H in the concrete mixture. On the other hand, (C-H) is a stable compound comparatively lower than C-S-H, leading to the increment of alkaline

state in the hardened concrete playing a significant role in the protection of the rebar available in the reinforced concrete against corrosion.

It can be seen that C_3S along with H_2O rapidly participates in the chemical reaction so that the major strength of the concrete obtained at the initial ages is due to the chemical composition of H_2O molecule with C_3S . Additionally, C_3S has the capability to produce remarkable heat; hence, it can relatively be known as a set-accelerating compound with moderate heat. About C_2S , it is urgent to point out that the C_2S properties are in converse with C_3S , which means that the initial set of C_2S is somewhat low, participating gradually in the chemical reaction so that the concrete strength at 7 days is more likely due to the reaction between C_2S and H_2O molecule. This combination generates a little amount of heat at the setting time, which can be considered as a set-retarding combination with low heat.

In the case of other hydration reactions, it can be named to the reaction between H_2O and aluminates in the cement compounds such as $C_3A(3CaO \cdot Al_2O_3)$ and $C_4AF(4CaO \cdot Al_2O_3 \cdot Fe_2O_3)$, shown in Equations (4) and (5) as one of the determinate factors against sulphate attack on concrete, known as one of the distinctive differences between pastes' OPC and T2PC in their composition, which will be explained later in the next sections.



It is worthwhile to state that the hydrated products achieved from the above-mentioned relations, Equations (4) and (5), have no notable role in the improvement of compressive strength in concrete. Therefore, it seems that the valuable compounds of Portland cement, C_3S and C_2S , have the most significant factor in the development of concrete strength so that the initial strength of concrete at early ages is because of the C_3S reaction and, subsequently, the acquisition of high strength in the next weeks is due to the participation of C_2S in the hydration process. However, both C_3S and C_2S have roughly the same effectiveness in terms of concrete durability in the long-term period [11,12]. Additionally, Lach et al. [13] also drew attention to the fact that Aluminate Calcium cement containing 70% Al_2O_3 and 28% CaO has the highest compressive strength compared to those with 40% Al_2O_3 and 35.5% CaO [13]. The novelty of this study is the concurrent utilization of cementitious ingredients containing low calcium for both Portland paste (T2PC) and pozzolanic additive as PC replacement to improve concrete property, especially compressive strength under ambient conditions. Additionally, the efficacy of other factors such as heat and alkaline solution on the improvement of concrete strength and permeability of this concrete type was experimentally explored in this investigation.

T2PC is the kind of modified Portland paste with the inclusion of low Ca compared to OPC in which it has a slightly retarded setting and relatively high resistance against sulphates' attack as well. Based on the preliminary studies in this field, the amount of C_3S was declined by 20%. In contrast, the value of C_2S was enhanced around 1.5 times in comparison with paste type 1, leading to the decrement of setting time and low heat, while the long-term strength of concrete manufactured by type 2 did significantly not alter compared to type 1. Additionally, a decrease in the value of C_3A can be seen as comparable to type 1, which is able to increase the concrete strength against sulphate attack. Cement with a low content of C_3A has been identified as sulphate-resistant cement, such as T2PC. Indeed, the sulphate attacks in concrete are accompanied by chemical reactions, which create the differential volume expansion in the hardened concrete, causing the compressive stresses, which are formed in several points of concrete in which the increment of these stresses leads to cracking and, eventually, failure in the concrete structure. The mechanism of these reactions can be assumed in one of the two following modes.

- The reaction between the sulphate ions and C_3A compound in the cement content is based on Equation (6), which will be faced with the volume enhancement by 220%.



The product of Equation (6) is called calcium sulfoaluminate, which is a colorless or pale-yellow crystal, and S is sulphur anhydride (SO_3) in this relation.

- The chemical reaction of sulphate ions and calcium hydroxide formed in the cement hydration will be exposed with a 120% volume increase.

It is necessary to point out that the method to control the sulphate attack is not only categorized in the use of T2PC or Type 5 Portland Cement (T5PC). Alternatively, the value reduction of calcium hydroxide in concrete using Pozzolanic materials, for instance, FA, is replaced as the percentage of Portland Cement (PC) in concrete content. Besides the use of compatible cement to neutralize the sulphate attacks, it is important to consider the water-to-cement (w/cm) ratio in a certain range, in which these supplementary limitations can increase the permeability of concrete in the adjacency of sulphate ion, resulting in the enhancement of concrete durability against environmental factors [14–18].

2. Methodology and Materials

The current study focused on the compressive strength of conventional concrete (T2PC) with a portion of fly ash as a PC substitute, as well as efficient parameters playing major roles in improving the mechanical properties of this concrete type. The following objectives were investigated.

- The assessment of compressive strength of GPC and conventional concretes throughout time.
- The influence of environmental factors (high temperature) on the formation of GPC concretes with the inclusion of T2PC and FA.
- A comparison of various concrete characteristics for GPC and PPC (workability/setting time/elasticity modules/tensile strength/flexural strength).
- A comparison of GPC and PPC water absorption (permeability).
- Determination of the optimal mix design for concrete mixes through comparing the dosages of FA and PC between the current experimental program and previous studies.

2.1. Materials

Low-calcium FA, known as 450-N (fineness category N, loss on ignition (LOI) Category B) based on BS EN 450-1 [19–21], was used in this research. The percentage of LOI subjected to fly ash category B was in the range of 2–7%, in accordance with BS EN 450 standard [22]. Indeed, LOI is commonly used as an indicator to identify the residual carbon content in fly ash. Additionally, % LOI indicates the presence of carbonates and combined water in clay minerals [23,24]. This FA type is widely employed in the UK, which can increasingly be found as the substantial ingredient in the low-carbon blended cement such as T2PC [25]. Indeed, 450-N FA used in this laboratory work has a higher value of siliceous and aluminous materials, compatible with other cementitious components, in which it can combine with liberated lime during the cement hydration in the presence of H_2O . The details of the chemical composition of FA along with other requirements in accordance with EN 450-1 are presented in Table 1. The CEM III/A-L 32.5 R in accordance with BS EN 197-1 [26,27] was considered for this study [28]. Further details subjected to the chemical, mechanical, and physical requirements for this type of Portland paste are exhibited in Table 2.

Table 1. Requirements for Fly ash (EN 450-1, 2012).

Property	Unit	Requirement Based on EN 450-1: 2012
Loss on ignition for Category B	% By mass	2.0–7.0
Water Requirement	%	≤95
Fineness Fraction for Category N ≥ 45 μm	% By mass	≤40
Soluble Phosphate (P ₂ O ₅)	% By mass	≤100
Total Phosphate	Mg/kg	---
Initial Setting	Min	2
Sum SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃	% By mass	≥70
Reactive SiO ₂	% By mass	≥25
Reactive CaO	% By mass	≤10
Sulphate (SO ₃)	% By mass	≤3
Free Calcium Oxide	% By mass	2.5
Soundness	Mm	≤10
Magnesium Oxide (MgO)	% By mass	≤4.0
Chloride (Cl ion)	% By mass	≤0.10

Table 2. Chemical, Mechanical, and Physical Requirements for CEM II/A-L 32.5 R (BS EN 197-1, 2000).

Clinker [1]	Blast Furnace [2]	Silica Fume	Fly Ash		Limestone (CaCO ₃) [5]		Minor Additional Constituents	Strength Class			Initial Setting Time (min)	
			Siliceous [3]	Calcareous [4]	L [6]	LL [7]		32.5 R [8]				
								Compressive Strength (MPa)				
								Early Strength		Standard Strength		
2 Days	7 Days	28 Days										
80–94	0	0	0	0	6–20	0	0–5	≥10	0	≥32.5	≤52.5	≥75

[1]: Portland cement clinker is made by sintering a precisely specified mixture of raw materials (raw meal, paste, or slurry) containing elements, usually expressed as oxides, CaO, SiO₂, Al₂O₃, Fe₂O₃, and small quantities of other materials. [2]: Granulated blast furnace slag shall consist of at least two-thirds by mass of the sum of calcium oxide (CaO), magnesium oxide (MgO), and silicon dioxide (SiO₂). The remainder contains aluminum oxide (Al₂O₃) together with small amounts of other compounds. The ratio by mass (CaO + MgO)/(SiO₂) shall exceed 1. [3]: Siliceous fly ash is a fine powder of mostly spherical particles having pozzolanic properties. It consists essentially of reactive silicon dioxide (SiO₂) and aluminum oxide (Al₂O₃). The remainder contains iron oxide (Fe₂O₃) and other compounds. [4]: Calcareous fly ash is a fine powder, having hydraulic and/or pozzolanic properties. It consists essentially of reactive calcium oxide (CaO), reactive silicon dioxide (SiO₂), and aluminum oxide (Al₂O₃). The remainder contains iron oxide (Fe₂O₃) and other compounds. [5]: The calcium carbonate (CaCO₃) content calculated from the calcium oxide content shall be at least 75% by mass. [6,7]: The total organic carbon (TOC) content, when tested in accordance with prEN 13639:1999, shall conform to one of the following criteria: LL: shall not exceed 0.20% by mass; L: shall not exceed 0.50% by mass. [8]: In CEM II/A-L 32.5 R code, II is the main cement type: A indicates the proportion of cement clinker: A (higher), B (medium), and C (lower); L indicates the second main constituent, in this case limestone (CaCO₃); 32.5 is standard strength class; and R indicates rapid (higher) early strength.

SH pellets (Figure 1a) along with sodium silicate powder with the density of 2.13 gr/cm³ (20 °C) and further details related to the chemical composition of this liquid are given in Table 3. Coarse aggregate (CAG) (20 mm and 10 mm) and fine aggregate (FAG) (Figure 1b) were blended with diverse dosages of FA between 0–100% in this study (Figure 1c). The percentage of aggregates incorporated in the concrete mix design proposed in this study was 43%, 22%, and 35%, respectively. Further details of the mixed proportion of this investigation are exhibited in Table 4. A labelling system was considered to present the concrete mixes. Each mix was assigned four characters. The first two letters represent the inclusion of Portland and FA in a concrete compound. Subsequently, the following two numbers represent the percentage of each cementitious material incorporated in the concrete mixtures. For example, PF9010 means the binder is comprised of 90% T2PC

and 10% FA, respectively. For mix proportions involving 30%, 50%, and 70% fly ash, the experimental program was conducted in two categories with/without an alkaline solution to assess the role of these chemical dissolutions on the improvement of the mechanical properties of the concrete mixture used in this research.



Figure 1. (a) Alkaline Solutions, (b) Aggregates, (c) Fly Ash.

Table 3. Chemical Composition of Sodium Hydroxide (NaOH).

Assay	Min. 98%
Heavy metals (as Pb)	Max. 5 ppm
Total N (Nitrogen)	Max. 5 ppm
CO ₃ (as Na ₂ CO ₃)	Max. 1.0%
Cl (Chloride)	Max. 50 ppm
PO ₄ (Phosphate)	Max. 20 ppm
SiO ₄ (as SiO ₂)	Max. 100 ppm
SO ₄ (Sulphate)	Max. 50 ppm
Al (Aluminium)	Max. 10 ppm
Fe (Iron)	Max. 10 ppm
K (Potassium)	Max. 0.1%
Pb (Lead)	Max. 5 ppm

Table 4. Mix Proportion of Alkaline Solutions.

Mix	NaOH (gr)	Na ₂ SiO ₃ (gr)	Water (mL)	Na ₂ SiO ₃ /NaOH	NaOH Molarity (M)	Alkaline Solution/FA (By Mass)
PF7030A	250	625	1500	2.5	4.16	0.25
PF7030B	250	625	1500	2.5	4.16	0.25
PF5050B	428	1070	1500	2.5	7.13	0.25
PF3070B	600	1500	1500	2.5	10	0.25
PF00100	830	1600	1500	2.5	12.96	0.25

The usage of High-Volume Fly Ash (HVFA) concrete was initially recommended by Malhotra [29] at the Canadian Centre for Mineral and Energy Technology (CANMET) in the mid-1980s [23,29]. The concrete mix design is routinely utilized for this FA concrete containing more than 50% FA by weight of the entire cementitious materials and a w/cm ratio of less than 0.4 [23,30]. The concentration of the chemical solution was variable from 4.16 to 12.96 M (Table 4). For those mixes including sodium hydroxide, the ratio of w/cm was considered in the range of 0.25–0.6 (Table 5). Besides that, the ratio between alkaline solutions including sodium hydroxide and sodium silicate was constantly kept by 0.25 (Table 4). The specimens were demolded, sealed (to avoid moisture loss), and cured at room temperature (about 14–17 °C) after 24 h. Three samples from each experiment were submerged in a water bath under 21 °C until commencing with the compressive strength test at ages of 7, 28, and 90 days. This curing condition was analogous for all mix proportions except that the concrete mixture (PF7030A) was maintained under the curing temperature of 110 °C for evaluating the efficiency of heat on the development of concrete strength. Gelenium 51, provided by BASF company in the UK, was chosen as a superplasticizer (SP) in this study.

Table 5. Mixture Proportion of Experimental Concrete.

Mix	C _{Ag} 20 mm (Kg)	C _{Ag} 10 mm (Kg)	F _{Ag} (Kg)	FA (Kg) (%)	T2PC (Kg)	Water (L)	Superplasticizer (mL) (2.5%)	Curing Condition	w/cm	SS/SH	
1	PF10000	32.84	16.4	26.52	0 (0%)	12	7.2	0	Water Bath at 21 °C	0.6	-----
2	PF9010A	32.84	16.4	26.52	1.2 (10%)	10.8	7.2	0	Water Bath at 21 °C	0.6	-----
3	PF9010B	32.84	16.4	26.52	1.2 (10%)	10.8	6.6	30	Water Bath at 21 °C	0.55	-----
4	PF8020	32.84	16.4	26.52	2.4 (20%)	9.6	6	30	Water Bath at 21 °C	0.5	-----
5	PF7030A	32.84	16.4	26.52	3.6 (30%)	8.4	6	30	24 h heat (110 °C) 4–5 h keep at room temperature Water bath at 21 °C	0.5	2.5
6	PF7030B	32.84	16.4	26.52	3.6 (30%)	8.4	6	30	Water Bath at 21 °C	0.5	2.5
7	PF7030C	32.84	16.4	26.52	3.6 (30%)	8.4	6.6	30	Water Bath at 21 °C	0.55	-----
8	PF5050A	32.84	16.4	26.52	6 (50%)	6	6	30	Water Bath at 21 °C	0.5	-----
9	PF5050B	32.84	16.4	26.52	6 (50%)	6	5.5	30	Water Bath at 21 °C	0.45	2.5
10	PF3070A	32.84	16.4	26.52	8.4 (70%)	3.6	5.7	30	Water Bath at 21 °C	0.48	-----
11	PF3070B	32.84	16.4	26.52	8.4 (70%)	3.6	4	30	Water Bath at 21 °C	0.33	2.5
12	PF00100	32.84	16.4	26.52	12 (100%)	0	3	30	Room temperature (14–17 °C)	0.25	2.5

C_{Ag}: coarse aggregate, F_{Ag}: Fine aggregate, FA: Fly Ash, T2PC: Type II Portland paste, SS/SH: Sodium silicate to Sodium Hydroxide ratio.

2.2. Scanning Electron Microscopy (SEM)

SEM is commonly one of the most significant techniques for assessing the chemical and physical characterizations of fly ash utilized in concrete content [31]. As Postec et al. [32] noted, backscattered electron imaging (BSE), electron imaging (SEI), and EDS are the three most typical modes of operations in SEM analysis [32,33]. SEM analysis in the study by Barbara et al. [30] distinctly showed intermixing of Fe and Al-Si mineral phase along with the predominance Ca non-silicate materials in the case of class F fly ash as pozzolanic material with low Ca. About general chemical and physical characteristics and elemental composition of this fly ash type, Kumar et al. [34] carried out SEM analysis on OPC and FA to explore the morphology of concrete specimens. SEM results demonstrated that OPC particles are more irregular compared to FA particles, which are in spherical shapes, as shown in Figure 2. In this study, it was decided to use FA containing low calcium after assessing the results achieved from SEM analysis.

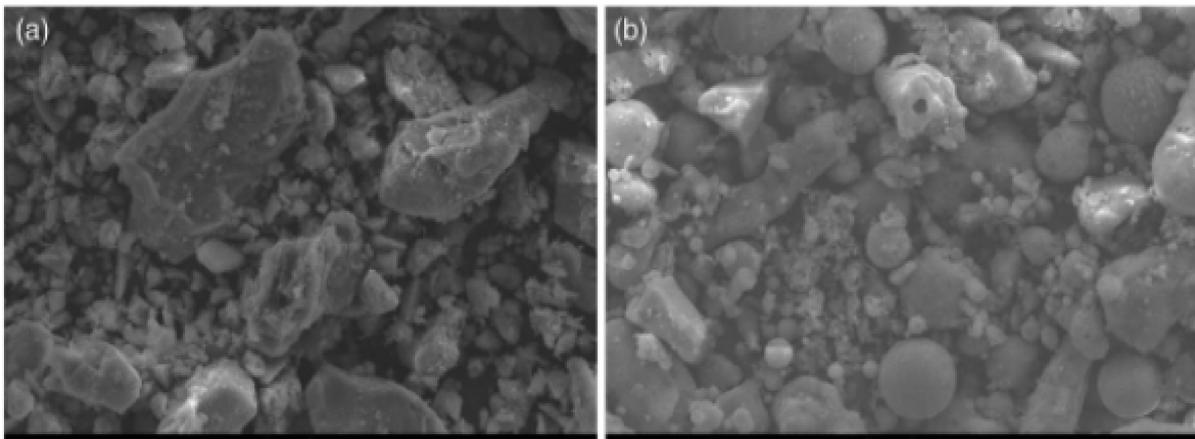


Figure 2. SEM of (a) OPC and (b) FA.

2.3. Experimental Design and Sieve Test

In this investigation, 36 cubes (three cubes for each concrete mix) were cast in the laboratory. Cubes (150 mm × 150 mm × 150 mm) were prepared based on a 1:2.21:4.10 mix design and a water–cement ratio in the range of 0.25–0.6 to compare the strength of cubes manufactured by diverse percentages of fly ash to investigate the influence of fly ash as a partial cement substitute on concrete strength at 7, 28, and 90 days. The fine aggregate was locally available river sand, which was passed through a 4-mm sieve, following BS 812103 standard [35]. The results of sieve analysis are given as follows in Table 6. The coarse aggregates were locally prepared, having two different sizes, which were passed through 10-mm and 20 mm-sieves. The grading table related to coarse aggregates of 10 mm and 20 mm are also presented in Tables 7 and 8, respectively.

Table 6. Sieve analysis for the fine aggregate of 4-mm size (sand).

Sieve Size (mm)	Mass Retained	Mass Passing	Weight Total	% Passing	BS Sieve Test Standard
8.0	0	166.63	166.63	100	100
6.3	0	166.63		100	95–100
4.0	3.99	162.64		97.61	85–99
1.0	35.76	126.88		76.14	57–97
0.5	44.53	82.35		49.42	30–70
0.25	62.33	20.02		12.01	0–40
0.063	19.66	0.36		0.22	0–4
Pan	0.36				

Table 7. Sieve analysis for the coarse aggregate of 10-mm size.

Sieve Size (mm)	Mass Retained	Mass Passing	Weight Total	% Passing	BS Sieve Test Standard
20.0	0	677	677	100	100
14.0	0	677		100	98–100
10.0	39	638		94.239	85–99
4.0	580	58		8.567	0–20
2.0	46	12		1.773	0–5
Pan	12				

Table 8. Sieve analysis for the coarse aggregate of 20-mm size.

Sieve Size (mm)	Mass Retained	Mass Passing	Weight Total	% Passing	BS Sieve Test Standard
40.0	0	1097	1097	100	100
31.5	0	1097		100	98–100
20.0	39	1058		96.445	85–99
10.0	1000	58		5.287	0–20
4.0	46	12		1.094	0–5
Pan	12				

2.4. Specimen Preparation and Concrete Manufacturing Process

As previously explained, the concrete mixtures consisted of CEMII (T2PC), fly ash (BS EN 450-N standard), CAg, FAg, alkaline liquid, water, and SP. Initially, three types of aggregates, along with T2PC and FA, were quantified before mixing. Both CEMII and fly ash were prepared from CEMEX supplier in the U.K. To make chemical solutions, SH pellets and SS powders were separately blended with water in the $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio of 2.5 to produce an alkaline liquid with different molarities. This liquid was provided 24 h before mixing day. The alkaline solutions should be mixed, which causes the polymerization to be formed more easily, based on the Davidovits method [36].

Generally, the manufacturing process shown in Figure 3 can be categorized into three steps.

Step 1: All solid materials were mixed around 3 min after quantifying by concrete mixer. The value used was considered based on the calculation of the number of specimens used in this part of laboratory work.

Step 2: The alkaline solutions previously prepared (Figure 1a) within 24 h before blending materials were poured over the solid materials for the mixes of PF7030A, PF7030B, PF5050B, PF3070B, and PF00100 containing fly ash as the replacement of Portland cement by 30%, 30%, 50%, 70%, and 100%, respectively. Consequently, they were mixed for approximately 4 min.

Step 3: Before casting fresh concrete, the workability was immediately measured through a slump test. Subsequently, based on BS 1881, the produced concrete was poured into the cube molds with the standard dimension of 150 mm × 150 mm × 150 mm and to reduce the voids of concrete by compacting action on the vibrating table around 1–2 min. After this period, for the mixtures of PF7030A, firstly, the specimens were exposed to the oven under the curing condition, which was 110 °C, and the samples were removed from the oven after 24 h. Subsequently, these specimens were kept at the usual temperature (room heat) between 14–17 °C for 4–5 h, and, eventually, the specimens were put in the water bath at the temperature of 21 °C until commencing the compressive tests at the age of 7, 28, and 90 days in this study.

As for the other concrete mixtures, the cube specimens were covered by a film to lessen water evaporation that happened over curing condition and were maintained in the molds for 24 h at room temperature (14–17 °C) to deter the drastic change of environment, which may happen over this period. Consequently, the specimens were taken out from the molds and were kept in a water bath until the strength test at the age of 7, 28, and 90 days (Figure 4).

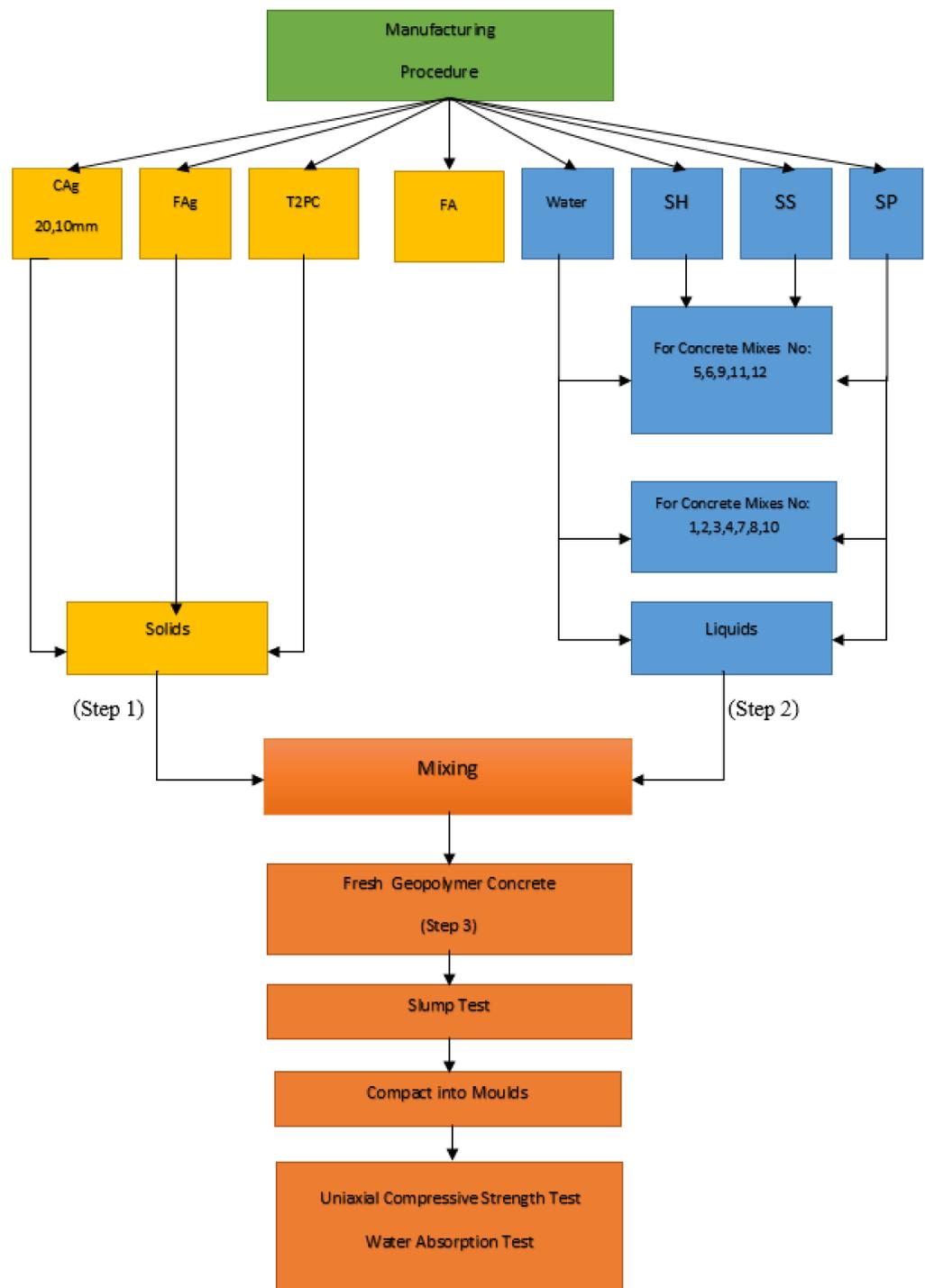


Figure 3. Geopolymer Concrete Manufacturing Process.

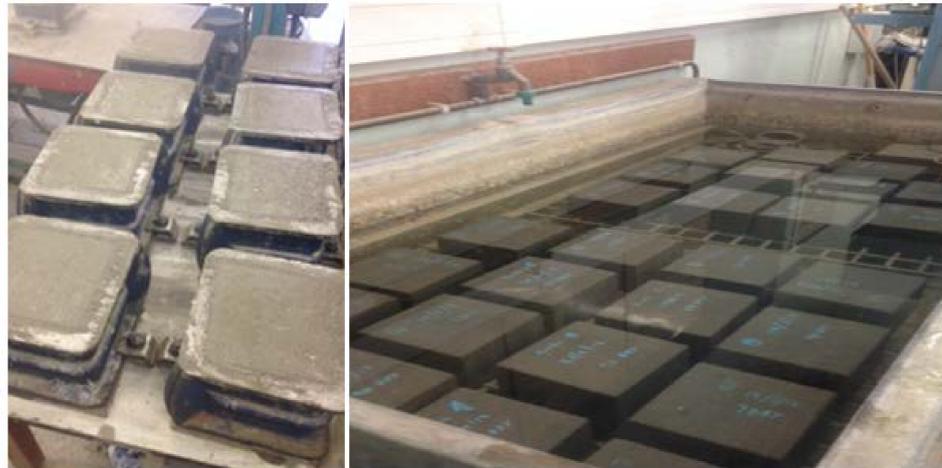


Figure 4. Specimen Preparation and Curing Conditions.

2.5. Test Procedure

In this investigation, the laboratory program was divided into two distinctive parts: The first part was about the comparison of the compressive strength of the concrete mixtures containing T2PC and different percentages of FA as the replacement of PC, and the second part was subjected to the behavior of concrete in terms of water absorption, considering the effect of diverse dosages of FA and the presence of alkaline solution to evaluate their performance in this case.

2.6. Compressive Strength

The test was carried out using an Avery Denison testing machine type 7226CB, calibrated in accordance with BS EN ISO 7500-1:2004 standard and is traceable to UKAS. This standard contains the determination of compressive strength of concrete cube specimens by applying a compressive axial load to molded concrete cubes at the rate of 5.7 kN/s until failure occurs. The compressive strength in all concrete mixtures was achieved by dividing the maximum load attained over the test with the cross-section area of the concrete samples. The concrete cubes of 150-mm width and 150-mm length, cured in the curing conditions mentioned above, were examined at the ages of 7, 28, and 90 days (Figure 4).

2.7. Water Absorption

In this study, a water absorption test was carried out based on ASTM C642 for the concrete cubes made of T2PC along with the different dosages of fly ash in the range of 10–70% by Portland cement mass at the ages of 7 and 28 days using Equation (7).

$$\text{Water Absorption (\%)} = \frac{W_a - W_d}{W_d} \times 100 \quad (7)$$

where W_a is the saturated sample weight in the air (gr) and W_d is the dried sample weight (gr).

3. Results and Discussion

3.1. Compressive Strength

The results of compressive strength were obtained from the average of three cube specimens tested for each mixture at the ages of 7, 28, 90 days, as shown in Figure 5. Additionally, the workability property through a slump test was conducted on the fresh concrete mixtures in this study. The results are summarized in Table 9.

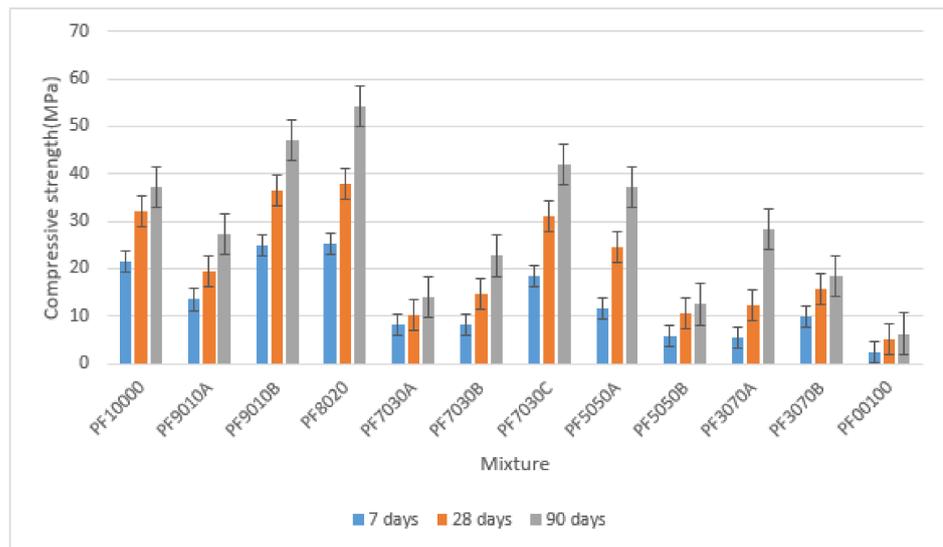


Figure 5. Compressive strength of concrete mixtures (T2PC-Fly Ash), (0–100% Fly Ash), with/without SP, with/without Alkaline Solution.

Table 9. Density, workability, and compressive strength of GPC concrete with diverse dosages of fly ash (0–100%) by Portland weight.

Mix	% T2PC	% Fly Ash	Average Dry Density (Kg/m ³)			Workability (mm)	Average Compressive Strength (MPa)			
			7 Days	28 Days	90 Days		7 Days	28 Days	90 Days	
1	PF10000	100	0	2332.8	2322.9	2322.46	70	21.56	31.93	37.13
2	PF9010A	90	10	2255.26	2248.8	2256.73	140	13.54	19.43	27.29
3	PF9010B	90	10	2367.86	2385.13	2385.2	22	24.99	36.63	47.10
4	PF8020	80	20	2356.5	2349.1	2339.23	30	25.24	37.84	54.11
5	PF7030A	70	30	2284.4	2281.4	2266.13	22	8.28	10.43	13.93
6	PF7030B	70	30	2314.53	2322.9	2307.1	22	8.14	14.58	22.73
7	PF7030C	70	30	2350.6	2351.56	2353.03	40	18.44	31.03	41.83
8	PF5050A	50	50	2350.56	2353.56	2371.3	40	11.68	24.53	37.07
9	PF5050B	50	50	2303.16	2351.03	2293.26	120	5.97	10.55	12.52
10	PF3070A	70	30	2349.6	2368.36	2351	45	5.49	12.33	28.43
11	PF3070B	70	30	2283.93	2335.76	2329.86	45	10.02	15.63	18.52
12	PF00100	0	100	2251.43	2173.8	2173.76	140	2.35	5.05	6.35

The slump values were seen to be decreased while increasing in FA up to 30%. The values of concrete strength were found to be enhanced at all ages with the increment of FA up to 20% as the replacement of T2PC, and beyond that, it steeply declined. The optimum value of 54.11 MPa was observed for the cubic specimens with 20% of FA and 80% of T2PC at 90 days. The increase in compressive strength with the inclusion of 20% of FA was probably due to the additional chemical reaction of FA with the presence of C-H molecules acquired from the hydration process of PC and H₂O, leading to the formation of calcium silicates in which the behavioral performance of C-S-H was simply explained in Section 1. The increase in strength values after 7 days was significantly seen for the mixtures with the inclusion of FA in the range of 0–70% as T2PC replacement without alkaline solutions. For example, in the mixtures with 20% FA after 7 days, the increase in strength values for 28 and 90 days was approximately 50.2% and 114.1%, respectively. This trend was

unlike the concrete mixtures containing 70% FA and alkaline solutions (PF3070B), where an increase of about 56–85% can be expected. It can be noted that this compressive strength development will happen as long as sufficient particles are present in the concrete content. Obviously, the deficiency of each chemical molecule such as calcium (Ca), silicate (SiO_3), and hydrate (H_2O) can have a preventive role for the formation of a polymeric chain of C-S-H, resulting in disturbance of the balance between the particles forming the chain. This factor can be justifiable in the reduction of the compressive strength of concrete mixtures including fly ash higher than 20% without using an alkaline solution.

As for the compressive strength of the mixes in which FA and alkaline solutions are concurrently used in concrete, it can be expected that the calcium ion can accelerate hardening and dissolution by providing nucleation in concrete. However, the evolution of concrete strength resulting from the concrete mixtures with/without alkaline liquid indicates that the higher values of compressive strength belong to the concrete binders that have no alkaline solutions in their content up to 50% of FA, whereas the concrete mix involving 70% of FA and SH with SS/SH ratio of 2.5 has a higher compressive strength than that of a concrete mix with 70% of FA and without an alkaline solution. However, the obtained results for the range of 30–70% FA for the concrete mixes with/without alkaline solutions reflect a steep reduction for compressive strength in which the following reasons can be considered for this phenomenon.

Generally, silica and alumina as the main compounds of FA have a convertible relationship with the amount of calcium incorporating in T2PC. However, the value of limestone (CaCO_3) in T2PC is between 6–20%, showing the low amount of calcium in this cement type, as shown in Table 2. Hence, the inclusion of FA exceeding 20% dosage would be exposed to the reduction of calcium as FA contains high silica and alumina and low calcium compared to T2PC. Thus, at higher FA content, calcium is less available for polymerization and results in less formation of polymerization products such as C-A-S-H and C-S-H, as explained in Section 1. This downward trend of compressive strength with certain cementitious materials was previously observed in other studies as well [37,38]. It seems that the role of C-S-H and C-A-S-H is more effective than N-A-S-H in this concrete type, given that the availability of calcium with the inclusion of T2PC and FA is decreased, while increasing the FA dosage in the concrete, leading to the reduction in the formation of C-S-H and C-A-S-H as these two polymeric chains, achieved an additional hydrate product, which co-exists with N-A-S-H as additional phases [39–41].

When comparing the results of this investigation to those of previous studies, it is essential to point out that the values of compressive strength at all ages were gradually increased due to OPC replacement by 20%, and, exceeding that, this trend was reduced. For instance, in a study by [42], the highest value of 66.81 MPa compressive strength was obtained for the specimens with 20% OPC as FA replacement at 365 days due to further reaction occurring between OPC and the use of alkaline solutions in this research. C-A-S-H and C-S-H were achieved as an additional hydrated product that has coexistence with N-A-S-H at additional phases. In contrast, the enhancement of OPC beyond 20% as FA replacement led to an increase in calcium content, and, consequently, a reduction of silica and alumina values was due to high calcium amount and low silica and alumina in OPC [43–46]. Therefore, a high value of calcium along with a lesser amount of silica and alumina, which are available in the chemical proportion of FA, was unable to produce N-A-S-H polymerization due to the lack of sufficient alumina and silica in the case of concrete mixtures with pozzolanic materials exceeding 20% FA.

3.2. Water Absorption

In this study, water absorption as the porosity of concrete was investigated at 7 and 28 days, as presented in Figure 6. It was observed that this concrete property considerably reduced with the inclusion of FA as replacement of T2PC up to 20%, and beyond that, it was dramatically enhanced. The least amount of water absorption was observed as 0.5% for the mixture containing 20% of FA at 28 days. Accordingly, the least value subjected

to this property was obtained as 0.79% at 7 days for concrete specimens with 20% of FA. Further details in terms of experimental work in this field are also shown in Table 10. It was also deduced that the downward trend was seen for all concrete mixtures at 28 days in comparison with those at 7 days. The laboratory observations indicated that the decrement in water absorption values with age development was not significant. However, a very marginal reduction in water absorption value was seen after 7 days, attributed to an ignorable change in microstructure and pore refinement that appeared after the initial ages of concrete improved by a slight enhancement in compressive strength after 28 days [47]. This improvement at the age of 28 days for the same specimens was not important as most polymerization mechanisms involving polymerization reactions along with the hydration process happened at the initial ages of concrete and, thereafter, a marginal modification can be notified [42].

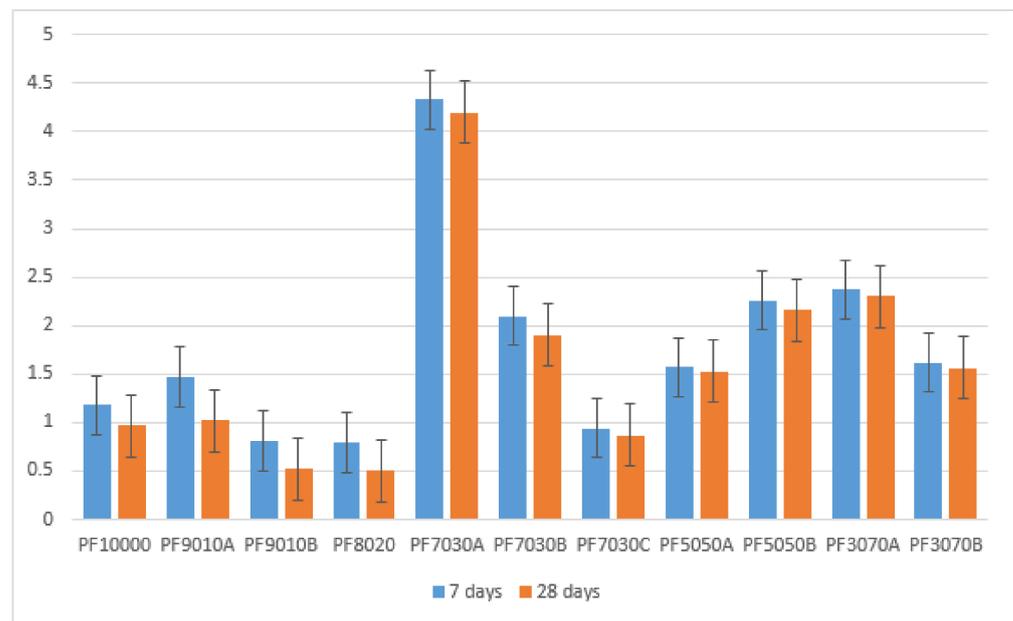


Figure 6. Water absorption percentage of T2PC with FA as a partial replacement at 7 and 28 days.

Table 10. Water absorption percentage for concrete mixtures with diverse dosages of FA (0–70%).

Mix	NaOH (Kg)	Na ₂ SiO ₃ (Kg)	Compressive Strength (MPa)		% Water Absorption		Alkaline Solution/Fly Ash
			7 Days	28 Days	7 Days	28 Days	
PF10000	0	0	21.56	31.93	1.18%	0.97%	N/A
PF9010A	0	0	13.54	19.43	1.47%	1.02	N/A
PF9010B	0	0	24.99	36.63	0.81%	0.52%	0.25
PF8020	0	0	25.24	37.84	0.79%	0.5%	0.25
PF7030A	0.25	0.625	8.28	10.43	4.33%	4.2%	0.25
PF7030B	0.25	0.625	8.14	14.58	2.1%	1.9%	0.25
PF7030C	0	0	18.44	31.03	0.94%	0.87%	N/A
PF5050A	0	0	11.68	24.53	1.57%	1.53%	N/A
PF5050B	0.428	1.070	5.97	10.55	2.26%	2.16%	0.25
PF3070A	0	0	5.49	12.33	2.37%	2.30%	N/A
PF3070B	0.600	1.5	10.02	15.63	1.62%	1.56%	0.25

Additionally, the highest water permeability was achieved as 30% of FA under the heated curing at 110 °C for 7 days. It revealed that these results are appropriate, in accordance with preliminary investigations reporting that curing GPC under the longer periods (18 h and 24 h) at elevated temperatures (e.g., 90 °C) led to a lower compressive strength. Andrade [48], Nadelman et al. [49], and Noushini and Castel [50] drew attention to heat curing for the concrete containing Portland pastes as the main cementitious materials enhancing the porosity of this concrete type compared to the ambient curing condition.

As for the permeation properties in specimens with high FA (>50%), the experimental results showed that the water absorption of these concrete mixes was higher than that of Portland cement concrete (PCC) due to the significantly higher volume of voids identified in the specimens with the inclusion of high FA. This was attributed to the formation of the gel type (NASH) in a binder [51]. The NASH ($\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$) as the main microstructure of a FA-based geopolymer had less density in comparison with the CSH ($\text{CaO}-\text{SiO}_2-\text{H}_2\text{O}$) in PCC [52]. Additionally, Provis et al. [53] pointed out that the presence of Ca inducing the presence of more bound water in the mixture was capable to provide greater pore-filling capacity to this gel type than to the NASH gel in geopolymer concrete. Overall, the results of water absorption for concrete specimens involving FA content exceeding 50% obtained in the current study were consistent with what has been found in previous studies. Naik [54] drew attention to the point that, beyond 91 days, concrete blended with 50% fly ash ($w/c = 0.35$) has a lower water permeability than Portland cement concrete [54]. Additionally, Yu et al. [55] expressed a similar view that the water permeability of mixtures with fly ash with dosages of 30% and 50% as PC replacement was higher than that of pure PC paste, and, after 180 days, PC paste blended with fly ash had a lower water permeability coefficient compared to PC paste without FA content.

As regards porosity, Kearsley and Wainwright [56] also drove the empirical formula expressing the relationship between GPC porosity and dry density after conducting several laboratory schedules that can be best described through Equation (8):

$$\rho = 18,700\gamma_d^{-0.85} \quad (8)$$

where ρ and γ_d are porosity (%) and dry density (Kg/m^3), respectively.

Table 11 and Figure 7 show a relationship between GPC porosity and dry density at 7 and 28 days of age based on Equation (8). The values of dry density were experimentally obtained, as shown in Table 9.

Table 11. The values of dry density and Porosity for each GPC mixture.

Mix No	FA Content (%)	Dry Density (γ_d) (Kg/m^3)		Porosity (ρ) (%)	
		7 Days	28 Days	7 Days	28 Days
PF10000	0%	2332.8	2322.9	25.65345	25.74635
PF9010A	10%	2255.26	2248.8	26.10125	26.0657
PF9010B	10%	2367.86	2385.13	25.33022	25.17424
PF8020	20%	2356.5	2349.1	25.43398	25.40206
PF7030A	30%	2284.4	2281.4	26.17471	26.1439
PF7030B	30%	2314.53	2322.9	25.82547	25.74635
PF7030C	30%	2350.6	2351.56	25.48823	25.47939
PF5050A	50%	2350.56	2353.56	25.4886	25.46098
PF5050B	50%	2303.16	2351.03	25.9338	25.48427
PF3070A	70%	2349.6	2368.36	25.49745	25.32568
PF3070B	70%	2283.93	2335.76	26.11928	25.62581

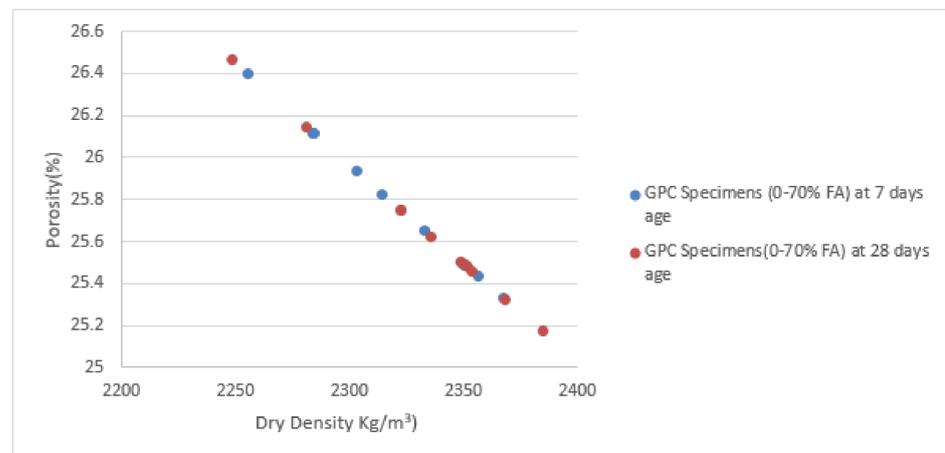


Figure 7. Relationship between porosity and dry density for GPC with the inclusion of FA in the range of 0–70% at 7 and 28 days of age.

Based on Figure 7, it can be concluded that there are no significant modifications between the amounts of porosity and dry density at 7 and 28 days of age, which are also in a similar trend with the results of water absorptions at ages of 7 and 28 days, previously discussed above. On the other hand, the comparison of findings subjected to water absorption (Table 10) and porosity (Table 11) demonstrated that the water absorption had a relative relationship with porosity so that the highest value for both water absorption and porosity belonged to the mixture involving 30% FA under 110 °C as the ambient cured GPCs in this study [48–50,56]. However, it seems that porosity is not the only factor influencing the water permeability of cement paste blended with fly ash, and further microstructure analyses are required in prospective studies [55].

4. Conclusions

The following observations were obtained based on the experimental results.

- The compressive strength of low-calcium, FA-based geopolymers was enhanced with the inclusion of FA as a replacement of T2PC up to 20% at all ages.
- The increment in concrete strength was observed due to additional calcium available in the chemical products formed by the hydration mechanism, which coexisted with the polymeric products of alumina-silicate.
- Although the use of fly ash with a percentage higher than 20% and alkaline solutions significantly enhanced the water absorption amount, the notable reduction of concrete strength was also observed, which was attributed to the relatively low balance in the value of ions in the chemical and hydration processes such as Na, Ca, Al₂O₃, and SiO₂. The ions were associated with the chemical combinations of T2PC participating in the concrete mixtures of this investigation.
- Concrete specimens including 30% of FA and alkaline solutions kept under the heat of oven at 110 °C for the initial 24 h as a curing condition (PF7030A) were found to have the highest values for both water absorption and porosity as well as relatively low compressive strength among all concrete mixtures at all ages. These results were comparable with previous studies described earlier in this field. Thus, the typical curing condition based on BS 1881 [57] is strongly recommended in terms of the concrete binders consisting of T2PC and low-calcium FA up to 30% as cement replacement, and, beyond that, further considerations such as the appropriate ratio of alkaline solution and using superplasticizers in which their chemical combinations are compatible with polymeric products are suggested for this purpose.
- Porosity is not the significant factor affecting the water absorption of Portland paste involving FA content. There is a relative relationship between water absorption and porosity; however, a little change was observed between 7 and 28 days for both water

absorption and porosity in each concrete mixture. Porosity is significantly dependent on dry density regardless of fly ash class (low or high calcium) and FA percentage in concrete content.

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