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Effect of incinerated paper sludge ash on fly ash-based geopolymer concrete

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Scientific Paper - Preliminary note

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The development of fly ash and incinerated paper sludge ash blend as a source material for preparing geopolymer concrete is presented in the paper. The specimens were prepared with varying percentage of fly ash replaced by paper sludge ash under different curing regimes. The compressive strength, splitting tensile strength, and bending strength values were tested and geopolymer microstructure was analysed. The up to 10% increase in strength of geopolymer concrete containing fly ash and paper sludge ash shows that good prospects exist for the use of this type of concrete for cast-in-situ applications.

Key words:
geopolymer concrete, fly ash, paper sludge ash, curing, compressive strength

Prethodno priopćenje

Senthamilselvi Pachamuthu, Palanisamy Thangaraju

Utjecaj pepela iz papirnog mulja na geopolimerni beton s letećim pepelom

U radu je prikazan razvoj veziva na bazi letećeg pepela i pepela dobivenog spaljivanjem papirnog mulja za pripremu geopolimernog betona. Uzorci za ispitivanje su pripremljeni s raznim postocima zamjene letećeg pepela pepelom iz papirnog mulja pri različitim uvjetima njege. Provedeno je ispitivanje tlačne čvrstoće, vlačne čvrstoće pri cijepanju i čvrstoće na savijanje te istraživanje mikrostrukture geopolimera. Povećanje čvrstoće geopolimernog betona s letećim pepelom i pepela iz papirnog mulja do 10 % pokazuje da postoje dobre perspektive za korištenje ovakvog tipa betona za ugradnju na licu mjesta.

Ključne riječi:
geopolimerni beton, leteći pepeo, pepeo iz papirnog mulja, njega, tlačna čvrstoća

Vorherige Mitteilung

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Auswirkung von Papierschlammasche auf Geopolymerbeton mit Flugasche

In der Arbeit wird die Entwicklung eines Bindemittels auf Basis von Flugasche und Asche aus der Verbrennung von Papierschlamm bei der Herstellung von Geopolymerbeton dargestellt. Bei den Prüfmustern wurden unterschiedliche Anteile von Flugasche durch Papierschlammasche bei diversen Instandhaltungsbedingungen ersetzt. Geprüft wurden die Druck-, Reiß- und die Verformungsfestigkeit; darüber hinaus wurde die Mikrostruktur des Geopolymers untersucht. Die Steigerung der Festigkeit von Polymerbeton infolge des Einsatzes von Flugasche und Papierschlammasche bis zu 10% bestätigt die guten Perspektiven für den Einbau dieser Betonsorte vor Ort.

Schlüsselwörter:
Geopolymerbeton, Flugasche, Papierschlammasche, Instandhaltung, Druckfestigkeit

1. Introduction

In 1978, Davidovits proposed that source materials rich in silicon (Si) and aluminium (Al), which are of geological origin, or rich in by-product materials such as fly ash (FA) and rice husk ash, can react with an alkaline liquid to produce binders. In 1979, he coined the term "geopolymer" to describe a new family of binder material [1].

Gaining high early strength is a characteristic of geopolymer concrete (GPC) when dry-heated or steam-cured, although ambient temperature curing is also possible [2]. Geopolymer binders can ensure performance comparable to traditional cementation binders in a range of applications with added advantage of significantly reduced greenhouse gas emissions [3]. Geopolymerisation is a reaction that chemically integrates minerals [4]. The exact mechanisms that govern geopolymerisation are still unclear [5].

Any material that contains mostly silicon (Si) and aluminium (Al) in amorphous form can be used as a source material for manufacturing geopolymer [5]. Quantitative prediction of the suitability of a specific mineral as source material is still not possible due to complexity of the reaction mechanisms involved [6]. Naturally available materials such as kaolin [7, 8], pozzolana [9, 10] and Malaysian marine clay [11], treated minerals such as metakaolin and waste materials such as fly ash [12-19], construction waste [20], red clay brick waste [21], fly ash and rice husk–bark ash [22], and fly ash and blast furnace slag [23], can be used. The chemical composition and particle size of these materials are important factors affecting geopolymer reaction.

Paper sludge ash (PSA) is generated from the incineration process of paper sludge, which is the largest by-product of the pulp and paper industry and is a major solid waste problem for the industry [24, 25]. The sludge that contains clay minerals such as kaolin is converted into metakaolin through incineration [26]. The produced PSA behaves as a pozzolanic material [27-29]. The sludge converted into a pozzolanic product on incineration can be used in the cement and concrete industries. The PSA comprises approximately 70 % – 80 % of amorphous silica and alumina [30]. This study examines the incinerated PSA as an alternative material that has good pozzolanic reactivity and that has been included in the geopolymer mix under different curing regimes.

2. Materials

2.1. Fly ash

Class F fly ash obtained from Mettur Thermal Power Station (Tamil Nadu, India) was used. The specific gravity of fly ash was 2.29. The loose bulk density of FA was about 748 kg/m^3 and its compacted value was about 1077.3 kg/m^3. Chemical properties of FA are given in Table 1. The scanning electron microscope (SEM) image of the fly ash is shown in Figure 1. The SEM image shows spherical shape of different-sized FA particles.

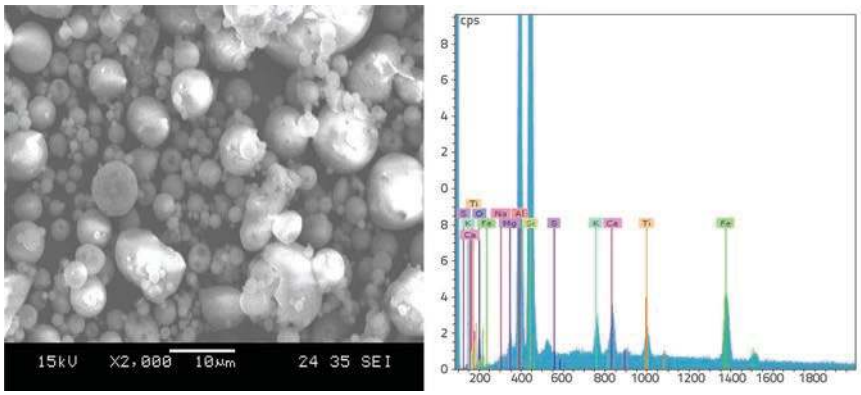

Figure 1. SEM and EDX images of fly ash

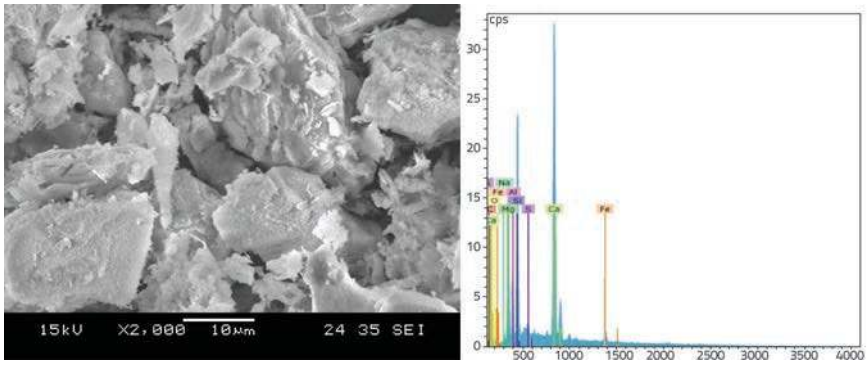

Figure 2. SEM and EDX images of paper sludge ash

2.2. Paper sludge ash

Paper sludge ash was obtained by incinerating the paper sludge obtained from Seshasayee Paper Mill (Pallipalayam, Erode, Tamil Nadu, India) at a temperature of 700 °C for 2 h. The specific gravity of the PSA was 2.3. The loose bulk density of the PSA was about 384.3 kg/m^3 and its compacted value was about 646.9 kg/m^3. The PSA was less bulky ccompared to FA. Chemical properties of the PSA are given in Table 1. The SEM image of the PSA is shown in Figure 2. The PSA particles are dominantly hexagonal and platy.

Table 1. Chemical composition of fly ash and paper sludge ash (mass %)

Composition	Fly ash	Paper sludge ash
SiO_2	53.97	35.25
Al_2O_3	34.66	7.09
Fe_2O_3	5.3	2.83
CaO	1.96	37.42
MgO	1.28	14.39
Na_2O	0.13	0.81
SO_3	0.25	0.69
K_2O	0.93	0.85
TiO_2	1.52	0.67
LOI	2.6	0.63

2.3. Applied aggregates

Locally available river sand confirming to grading zone II as per BIS 383-1970 was used as fine aggregate. Its specific gravity was 2.6 and fineness modulus was approximately 2.72.
Crushed blue granite stones 20 mm in maximum size were used as coarse aggregate. The specific gravity of this material was 2.86, and fineness modulus was approximately 7.27.

2.4. Alkaline activators

Alkaline activators used in this study consisted of the combination of sodium silicate and sodium hydroxide solution. Sodium hydroxide solution of 12M was prepared by diluting NaOH pellets of 99 % purity in water. The ratio of Na_2SiO_3/NaOH was kept as 2.5. Physical and chemical properties of NaOH and Na_2SiO_3 are given in Tables 2 and 3, respectively.

Table 2. Physical and chemical properties of sodium hydroxide

Appearance	White crystalline substance
Colour	White
Molecular weight	39.997 [g/mol]
Specific gravity	1.52
Assay	99 [%]
Carbonate (Na_2CO_3)	1 [%]
Chloride (Cl)	0.01 [%]
Sulfate (SO_2)	0.01 [%]
Lead (Pb)	0.002 [%]
Iron (Fe)	0.002 [%]
Aluminium (Al)	0.002 [%]

Table 3. The physical and chemical properties of sodium silicate

Appearance	Liquid (gel)
Colour	Light yellow
Boiling point	100 [°C]
Molecular weight	122.0632 [g/mol]
Specific gravity	1.53
Assay Na_2O	8.5 [%]
Assay SiO_2	28 [%]
H_2O	63.5 [%]

3. Experimental details

3.1. Mix composition

The GPC mix design was prepared for grade M35, which was obtained following the provisions of the Bureau of Indian Standards (BIS), code for ordinary Portland cement concrete, but with the replacement of cement with fly ash, and water/cement ratio with alkali activator solution to fly ash ratio.
To identify various mixes, letters A, B, and C were used for curing conditions: oven at 60 °C, external exposure, and ambient respectively. Each letter was provided with numeral 0, 5, 10, 15, and 20 to denote the percentage of fly ash replaced with PSA. The GPC mix proportion details are shown in Table 4.

3.2. Specimen preparation

The manufacture of GPC was conducted as per the procedure explained by Rangan [31]. Sodium hydroxide (in pellet form) was weighed to suit 12M and dissolved in water. The solution was well stirred for better dissolution and added to sodium silicate solution. Both solutions were mixed and kept in a container for 24 h before use. The materials were dry-mixed in a pan mixer for 3–4 min and alkaline solution was added and mixed for 4–5 min to make a uniform concrete mix.
The fresh GPC mixes were then filled in moulds and compacted by a table vibrator to expel air voids. Freshly cast specimens were given 1 day delay time before being subjected to their respective curing conditions.

3.3. Curing

In this study, three curing regimes were used to study the influence of curing temperature on the fly ash–based GPC with and without PSA. In the AC, concrete specimens were placed in a shaded area with a maximum temperature of 28 °C. These specimens were constantly protected from direct sunlight and rainfall until the testing days. The concrete specimens in the EEC were placed in an area exposed to direct sunlight that is at the same time protected from rain. The maximum temperature in

Table 4. Mixture proportion details

Mix code	Proportion [kg/m³]						Curing condition
	Fly ash	Paper sludge ash	Coarse aggregate	Fine aggregate	NaOH solution	Na_2SiO_3	
A0	425.73	0.00	1212.60	642.50	54.74	136.84	Oven at 60°C (OC)
A5	404.45	21.29	1212.60	642.50	54.74	136.84	
A10	383.16	42.57	1212.60	642.50	54.74	136.84	
A15	361.87	63.86	1212.60	642.50	54.74	136.84	
A20	340.59	85.15	1212.60	642.50	54.74	136.84	
B0	425.73	0.00	1212.60	642.50	54.74	136.84	External exposure (EEC)
B5	404.45	21.29	1212.60	642.50	54.74	136.84	
B10	383.16	42.57	1212.60	642.50	54.74	136.84	
B15	361.87	63.86	1212.60	642.50	54.74	136.84	
B20	340.59	85.15	1212.60	642.50	54.74	136.84	
C0	425.73	0.00	1212.60	642.50	54.74	136.84	Ambient (AC)
C5	404.45	21.29	1212.60	642.50	54.74	136.84	
C10	383.16	42.57	1212.60	642.50	54.74	136.84	
C15	361.87	63.86	1212.60	642.50	54.74	136.84	
C20	340.59	85.15	1212.60	642.50	54.74	136.84	

externally exposed method reached to 41 °C. For comparison purposes, oven curing (OC) at 60 °C exposure conditions was also conducted for the specimens. Freshly cast specimens were given 1 day delay time before being placed into the oven. After demoulding, the specimens were placed in an electrical oven and dried at the temperature of 60 °C for 24 h. After 24 h, the specimens were transferred to ambient conditions (AC).

3.4. Testing

Compressive strength tests were conducted on 150 × 150 × 150 mm cube moulds at 3, 7, and 28 days, whereas 100 × 200 mm cylindrical moulds were used for determining split tensile strength at 3, 7, and 28 days. Prisms 100 × 100 × 500 mm in size were used to determine flexural strength at 28 days. Microstructure properties of GPC were evaluated through SEM along with the energy-dispersive X-ray spectroscopy (EDX) analyses.

4. Results and discussion

4.1. Mechanical properties of GPC specimens

Development of mechanical strength in the hardened GPC is the basic performance indicator of the alternative source material because it provides a fundamental description of the quality of geopolymerization products. The compressive, tensile, and flexural strengths of concrete, determined at 3, 7, and 28 days, are given in Figures 3-9 for three curing conditions. An improvement in the compressive strength, tensile strength, and flexural strength was registered for the specimens with 10 % PSA replacement under EEC and AC conditions. The PSA-added mixes under oven curing conditions showed a decrease in strength compared to the mixes without PSA under the same curing conditions that provide maximum strength. The increase in compressive strength of GPC having PSA at AC conditions may be due to the pozzolanic reaction, which is more pronounced at low temperatures then at elevated ones due to the use of calcium compounds in the PSA material. The same fact was also established in [32], where it was discovered that the solubility of Al and Si increases significantly with temperature. However, this is not the case with Ca compounds.

It can nevertheless be observed that at 28 day the PSA-added GPC exceeded the target strength of 35 MPa and attained substantial strength of around 38 MPa for the 10 % addition under EEC and AC conditions.

The inclusion of PSA into the FA-GPC, and the curing temperature under different curing conditions, are the parameters that influence mechanical properties of GPC.

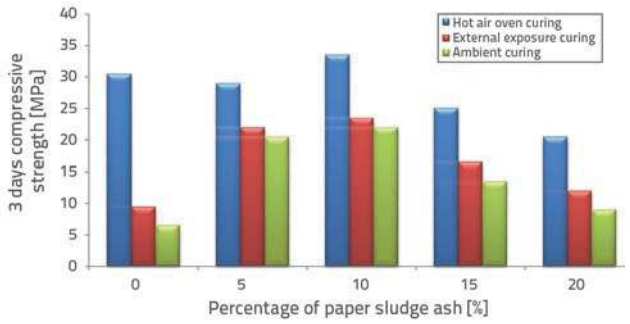

Figure 3. Compressive strength of FA-GPC and FA-PSA GPC after three days of curing

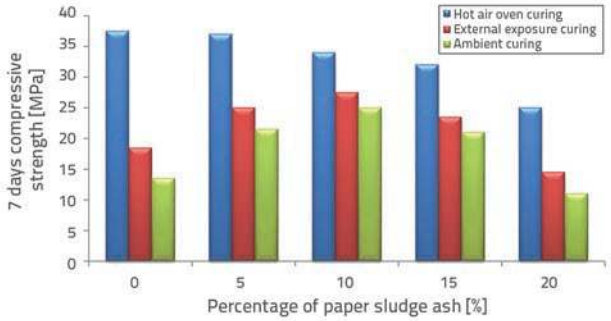

Figure 4. Compressive strength of FA-GPC and FA-PSA GPC after seven days of curing

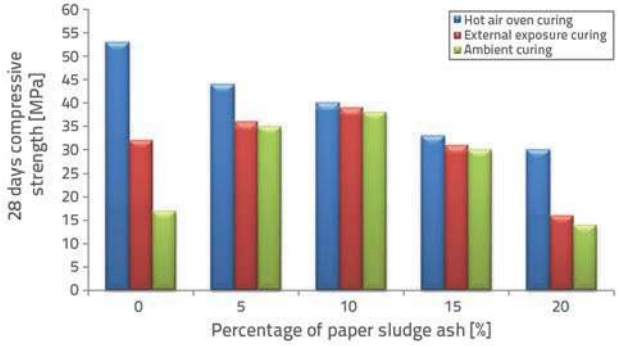

Figure 5. Compressive strength of FA-GPC and FA-PSA GPC after 28 days of curing

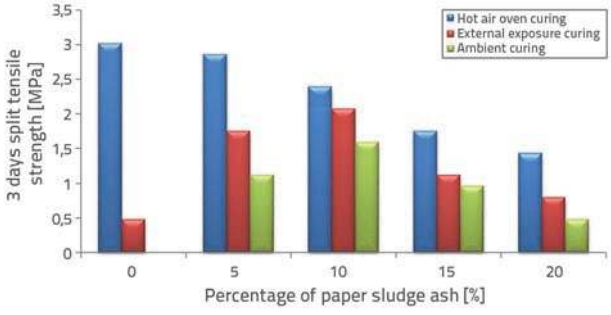

Figure 6. Split tensile strength of FA-GPC and FA-PSA GPC after three days of curing

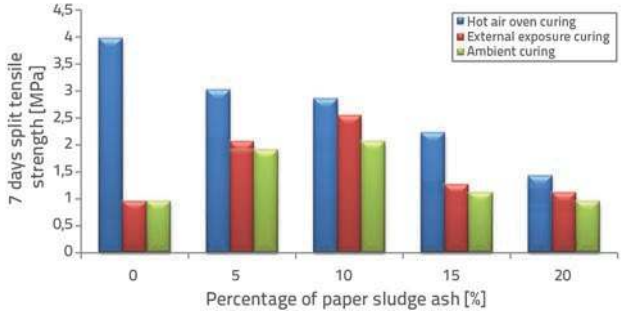

Figure 7. Split tensile strength of FA-GPC and FA-PSA GPC after seven days of curing

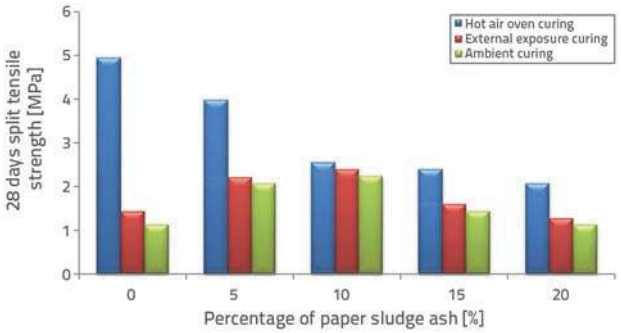

Figure 8. Split tensile strength of FA-GPC and FA-PSA GPC after 28 days of curing

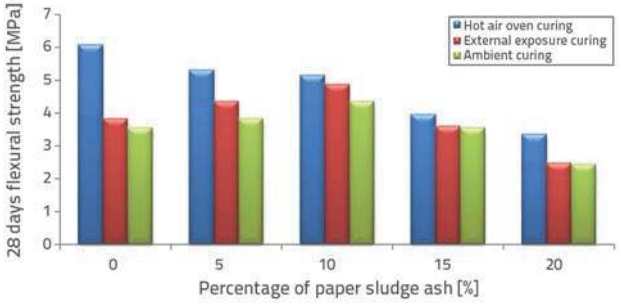

Figure 9. Flexural strength of FA-GPC and FA-PSA GPC after 28 days of curing

4.1.1. Effect of paper sludge ash addition to fly ash – based GPC

Fly ash–based GPC achieves its strength by geopolymerization in a highly alkaline environment. From the study results, it can be observed that an increase in PSA from 0 % to 10 % increases the compressive strength, tensile strength, and flexural strength in different curing conditions, except in the OC conditions. The strength development in FA-PSA GPC occurs because of the presence of CaO, which is more represented than SiO_2 in the chemical composition of PSA. This is the reason why the binding system of FA-PSA GPC became silica–calcium (Si+Ca) instead of silica–aluminium (Si+Al), as in FA-GPC.

The reaction mechanism of geopolymerization varies significantly depending on the presence of Ca in the geopolymer mixture [33]. The presence of Ca exerts a positive effect on mechanical properties of the geopolymeric binder, but the exact role of calcium during the geopolymerization process still remains unclear [34]. Calcium reacts with the soluble silicate and aluminate species, which leads to formation of the calcium silica hydrate and the calcium aluminate hydrate gel phase [35]. The compressive strength increases due to coexistence of the alkali aluminosilicate gel and calcium silica hydrate or the calcium aluminate hydrate gel [36]. Mechanical properties decrease beyond 10 % replacement of fly ash by PSA. The reason for this behaviour can be clarified by examining microstructure of the samples.

4.1.2. Curing mode

The rate of geopolymer gel production depends on the rate of dissolution and polycondensation, which varies with the difference in curing temperature. Under AC, fly ash–based GPC develops the lowest strength due to absorbance of an excessive amount of water from the solution, which hinders the polycondensation process and results in lower strength compared to other curing conditions. It is a known fact that hydroxide activity is significantly affected by the excessive water content in the GPC system [37].

The absorptive characteristic of PSA reduces water content in the GPC through the water absorption mechanism. Even at a lower gelation rate under AC condition, the addition of PSA contributes to an increase in compressive strength through reduction of the water-filled pores by the above mechanism. Because of PSA, calcium compounds in the geopolymer mix improve mechanical strength of samples cured at ambient temperature, but reduce the strength of samples cured at higher temperature due to possible existence of different hardening mechanisms [32, 38].

Under the EEC condition, specimen temperatures were slightly higher than those of specimens under AC condition. Hence, the improvement in strength is observed in both fly ash and fly ash-PSA-based GPCs.

Rapid dissolution and polycondensation of fly ash in the non-PSA-based GPC are completely supported by elevated temperatures. Thus, the maximum compressive strength of 53 MPa was achieved due to elevated temperature only. But inclusion of PSA in the OC condition adversely affected the strength performance of the fly ash–based GPC. The presence of calcium in PSA possibly does not allow formation of the three-dimensional geopolymeric network and consequently reduces mechanical properties [39].

4.2. Microstructure analysis of fly ash-PSA-based geopolymer concrete

Representative specimens for SEM (scanning electron microscope) and EDX (energy-dispersive X-ray spectroscopy) analysis were selected from each curing condition with the PSA (paper sludge ash) content of 0 % and 10 %.

4.2.1. Scanning electron microscope (SEM) analysis

Qualitative analysis of GPC microstructure was conducted in this study through the field-emission SEM investigations on FA-GPC and FA-PSA GPC with 10 % of PSA.

Figure 10.a shows the SEM image of geopolymer matrix in the fly ash–based system under hot air OC conditions. It looks sound and shows a dense, compact microstructure

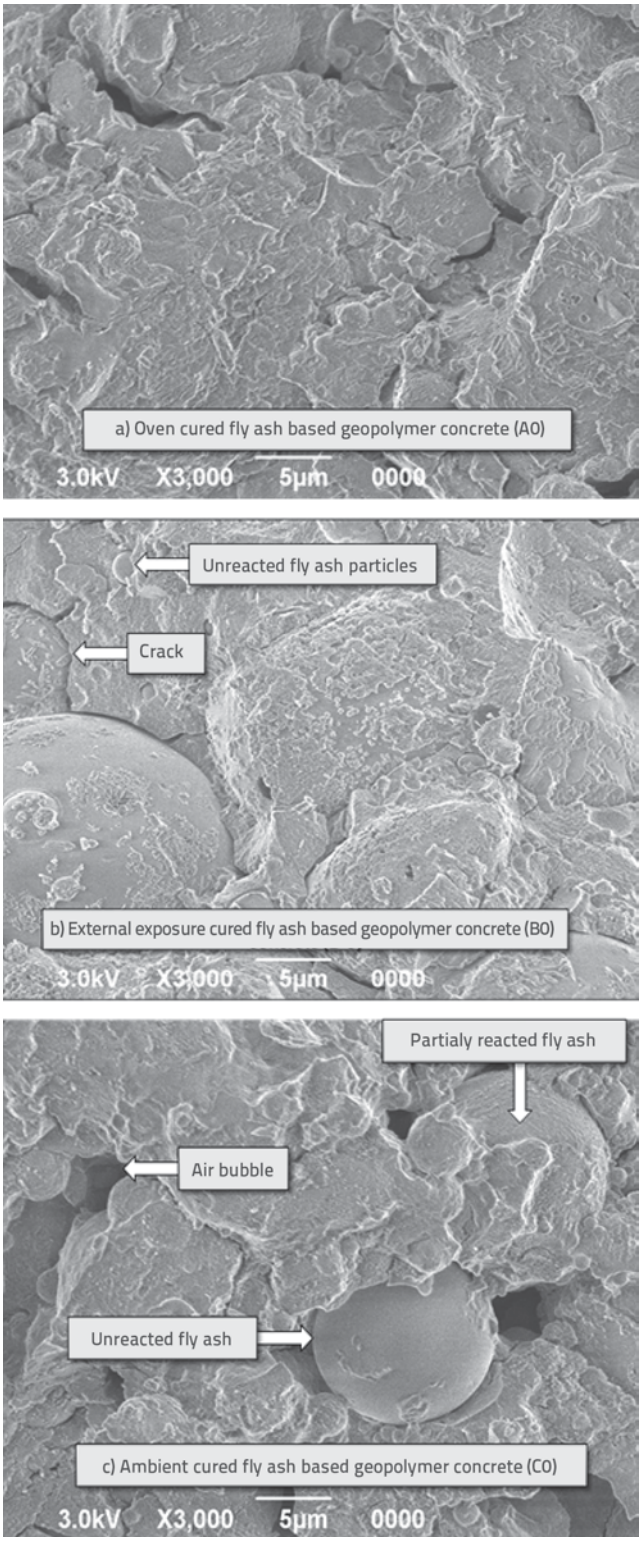

Figure 10. SEM images of fly ash–based geopolymer concrete specimens: a) SEM image of specimen A0; b) SEM image of specimen B0; c) SEM image of specimen C0

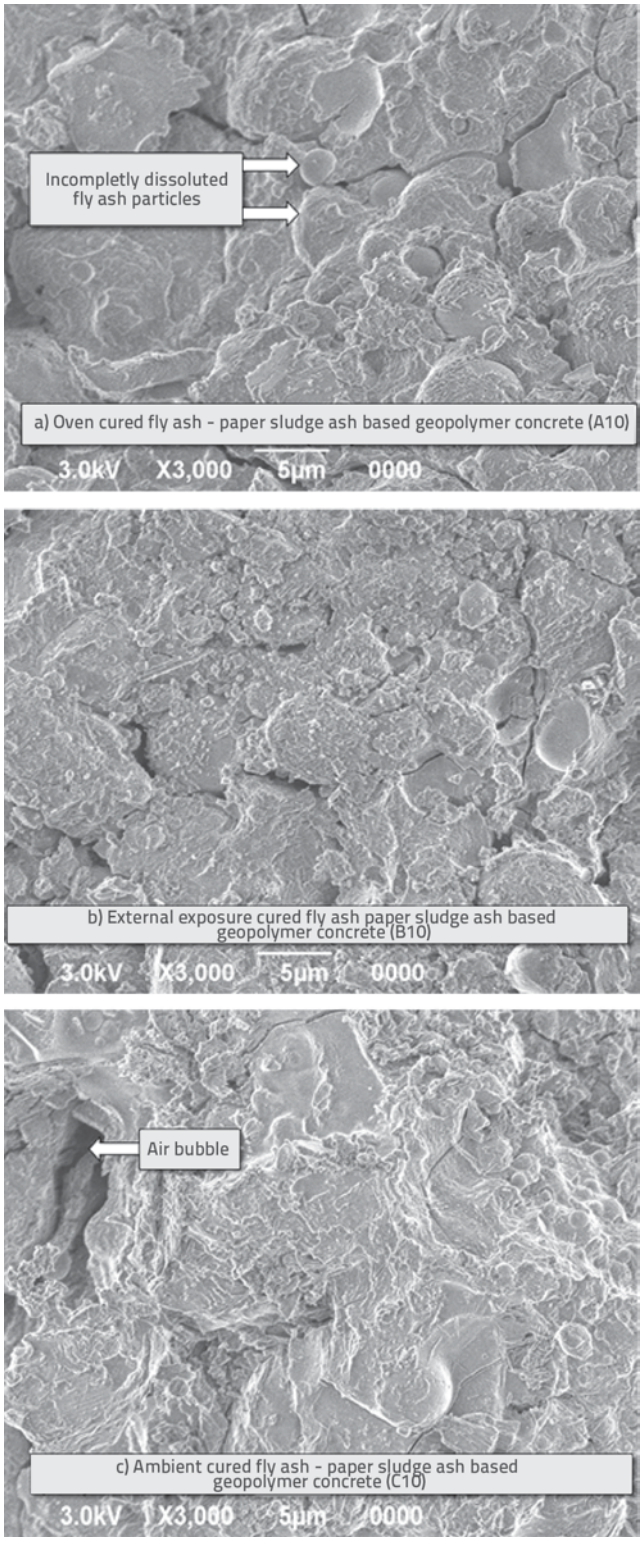

Figure 11. SEM images of fly ash-paper sludge ash–based geopolymer concrete specimens: a) SEM image of specimen A10; b) SEM image of specimen B10; c) SEM image of specimen C10

with a smaller proportion of unreacted raw materials in the sample. This is consistent with the compressive strength test results. Figure 11.a shows the SEM image of geopolymer matrix in the fly ash-PSA-based system under OC conditions, showing some unreacted or partially reacted fly ash particles. The FA-GPC cured under EEC and AC conditions shows a number of air bubbles and the partially or unreacted fly ash particles in its microstructure. It can be observed that the low-temperature curing does not promote a high dissolution rate of fly ash by the alkaline solution [40], which results in formation of a large proportion of unreacted and partially reacted fly ash particles in the system. Figure 11.b and 11.c shows that the homogeneity in the specimen microstructure is improved by addition of PSA under EEC and AC conditions compared to that in the non-PSA-added specimens under the same curing conditions (Figure 10.b and 10.c). In most of the SEM images, some cracks can be observed on the surface of the specimens, which might be due to mechanical damage during sample preparation.

4.2.2. EDX analysis

Concrete samples for EDX analysis were selected from each curing condition with PSA addition at 0 % and 10 %. The EDX analysis results for three curing conditions are presented in Table 5. It can be seen from these results that Si, Al, Na, Fe, Ca, and O appear to be dominant elements in the FA-PSA GPC specimens.

In OC, the Si/Al ratio detected is 3.12 for the FA-GPC, and 3.04 for the FA-PSA GPC specimens. The decrease in the trend of Si/Al ratio is due to chemical composition of PSA. When the curing temperature rises, the amount of calcium intensifies proportionally [28], which does not allow formation of a three-dimensional geopolymeric network [41]. The Si/Al ratio value may decrease due to rapid dissolution of the source materials. The EEC and AC specimens have an increasing Si/Al ratio of 2.947 and 2.783 for the FA-PSA GPC specimens, compared to FA-GPC specimens with Si/Al ratio of 2.506 and 2.1163, respectively. Dissolution of silica species from PSA particles contributes consistently to an increase in the Si/Al ratio trend for the FA-PSA GPC specimens.

5. Conclusion

The effect of adding PSA to the fly ash–based GPC on the mechanical and microstructure properties was investigated in this study. Oven-cured non-PSA specimens yielded maximum strength compared to the non-PSA samples under EEC and AC conditions. The addition of PSA in FA GPC decreases mechanical strength under OC conditions. Hence, the use of this material is not suitable under this curing condition. The addition of PSA in the FA GPC improves mechanical properties under both

Table 5. EDX element weight percentage analysis

Mix code	Element [wt %]				
	O	Si	Al	Na	Ca
A0	53,56	24,03	7,7	6,84	1,7
A10	52,91	24,49	8,05	7,82	2,55
B0	53,54	22,81	9,1	8,42	1,51
B10	52,02	24,64	8,36	7,46	2,56
C0	52,75	24,55	11,6	4,85	1,54
C10	54,35	23,8	8,55	7,59	2,27

EEC and AC conditions. Beyond 10 % addition of PSA, there is a decline in the mechanical strength under EEC and AC conditions. The characteristic of 35 MPa was achieved under all the three curing conditions of the fly ash-PSA-based GPC specimens. The results under AC and EEC conditions showed that geopolymer technology can be used in the cast-in-situ concrete construction. The test results were also confirmed by the SEM observation. It is evident from the EDX analysis results that mechanical properties of FA-PSA GPC are influenced by the Si/Al ratio.

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