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CHARACTERISATION AND STRENGTH ACTIVITY INDEX OF ECO-PROCESSED POZZOLAN

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ABSTRACT: Eco-processed pozzolan (EPP) is a newly emerging pozzolanic material produced from the extraction, refinement, and calcination of spent bleaching earth. Due to its pozzolanic characteristics, EPP is a novel interest in the material sustainability of concrete technology in Malaysia today. However, there is still a lack of studies on how much EPP replacement fulfills the minimum requirements of 75% of the value of the strength activity index (SAI) as specified in the ASTM C618 standard. This study investigated the chemical, physical, mineralogical, and microstructural properties of EPP. The results revealed that EPP fulfilled the criteria as a class N pozzolan as per ASTM C618. According to the XRD result, the mineralogical data suggest that the OPC has a high amount of alite and belite at 65.65% and 10.26%, respectively. In contrast, the EPP showed high amorphous silica at 64.28%. Based on the scanning electron microscopy (SEM) images, the EPP particles had a spherical and porous structure, which increased the amount of water required for the mortar containing EPP. This study also examined EPP's strength activity index (SAI) at 10%, 20%, 30%, 40%, 50%, and 60% as partial cement replacement. The results indicated that the SAI was reduced with high EPP replacement percentages due to lower CaO content. The 28-day SAI value satisfied the minimum requirement of pozzolan as per ASTM C618 for cement replacement by up to 40%. In conclusion, EPP performed well as a cementitious supplementary material, with 10% and 20% being optimal replacements for cement.

ABSTRAK: Pozzolan Terproses-Eko (EPP) merupakan bahan pozzolan yang baharu terhasil daripada proses pengekstrakan, penapisan dan pengkalsinan daripada tanah peluntur terpakai. Disebabkan ciri-ciri pozzolaniknya, EPP menjadi tarikan terbaru dalam teknologi bahan konkrit lestari di Malaysia hari ini. Namun, masih terdapat kekurangan kajian mengenai berapa banyak penggantian EPP yang dapat memenuhi keperluan minimum 75% nilai indeks aktiviti kekuatan (SAI) seperti yang dinyatakan di dalam ASTM C618. Kajian ini menyelidik sifat kimia, fizikal, mineralogi, dan mikrostruktur EPP. Dapatan kajian mendapati bahawa EPP memenuhi kriteria sebagai pozzolan kelas N mengikut ASTM C618. Menurut dapatan XRD, data mineralogi menunjukkan bahawa OPC mempunyai kandungan alite dan belite yang tinggi iaitu sebanyak 65.65% dan 10.26%, masing-masing. Manakala, EPP menunujukkan kandungan silika amorfus yang tinggi pada 64.28%. Berdasarkan imej pengimbas mikroskop elektron (SEM), zarah EPP mempunyai struktur sfera dan berliang, di mana ia meningkatkan jumlah air yang diperlukan untuk mortar yang mengandungi EPP. Kajian ini juga mengkaji SAI EPP pada 10, 20, 30, 40, 50, dan 60% sebagai penggantian separa simen. Dapatan kajian menunjukkan bahawa penurunan nilai SAI dengan peratusan

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penggantian EPP yang tinggi disebabkan kandungan CaO yang rendah. Nilai SAI pada 28 hari memenuhi syarat minimum pozzolan mengikut ASTM C618 bagi penggantian simen sehingga 40%. Kesimpulannya, EPP berprestasi baik sebagai bahan tambahan simen, dengan 10% dan 20% sebagai penggantian simen yang optimum.

KEYWORDS: *Eco-Processed Pozzolan, Strength Activity Index, Partial Cement Replacement, Water requirement*

1. INTRODUCTION

Concrete is a widely used construction material made of aggregates, water, and cement. However, cement production has a substantial adverse environmental impact, primarily due to high carbon dioxide (CO2) emissions released into the atmosphere during manufacturing [1]. One way to reduce the carbon footprint of construction is by partially replacing cement with pozzolanic materials, which can significantly improve the performance of concrete and reduce its cost [2-4].

Eco-processed pozzolan (EPP), also known as spent bleaching earth ash (SBEA) [5,6], processed SBEA [7], and calcined SBEA [8], is a newly emerging pozzolanic material and can be used as a cement replacement in concrete. EPP originated from bleaching earth, a pozzolanic mineral used in the production of palm oil. It is incorporated into the refining processes to convert crude palm oil into refined, bleached, and deodorised palm oil. Further extraction, refinement, and calcination of the SBE produce EPP [9,10]. The material was manufactured from a refining plant at Lahad Datu, Sabah.

Many studies have reported that EPP has a large silica (SiO2) content ranging from 42.19% to 60.4% [5-11]. This makes EPP a good pozzolanic material that can partially replace cement in concrete production. Abd Rahman et al. (2020) examined the pozzolanic reactivity of EPP using the strength activity index (SAI) test. The results showed that the SAI values of EPP at both curing ages, 7 and 28 days, were more than the minimum requirement of 75% according to the ASTM C618 standard [10]. This indicates that EPP has sufficient reactivity to qualify as a pozzolan.

Recently, there have been many studies on the use of EPP in regular concrete [12-15], geopolymer concrete [16], pavement block [17], and foamed concrete [7,18]. The studies have revealed that the rate of pozzolanic reaction to produce C-S-H gel increased due to high SiO2 content in EPP. This gel fills the voids in concrete, making the concrete denser, thus improving its strength. Furthermore, the inclusion of EPP in concrete also enhanced its resistance to sulphate, carbonation, and chloride attack [12-14].

Although many studies have been conducted on using EPP as a cement replacement, there is still a lack of studies on how much EPP replacement would fulfil the minimum requirement of 75% SAI value. Therefore, this research aims to examine the effect of incorporating 10, 20, 30, 40, 50, and 60% of EPP on the SAI of mortar.

2. METHODOLOGY

2.1. Material

The cement mortars in this study consisted of Ordinary Portland Cement (OPC), EPP, sand, and water (see Fig. 1). The OPC was the Cap Gunung brand from Cement Industries (Sabah) Sdn. Bhd., which complied with MS EN 197-1 CEM I 52, 5N [19]. Meanwhile, the EPP was collected from Eco Oils Sdn. Bhd. is based in Lahad Datu, Sabah.

The fine aggregate was local river sand sourced from Kota Belud, Sabah. The sand was dried in an oven at 105± 5ºC to remove moisture for 24 hours. Potable water, which complies with ASTM C1602 [20], was used to mix and cure the mortars.

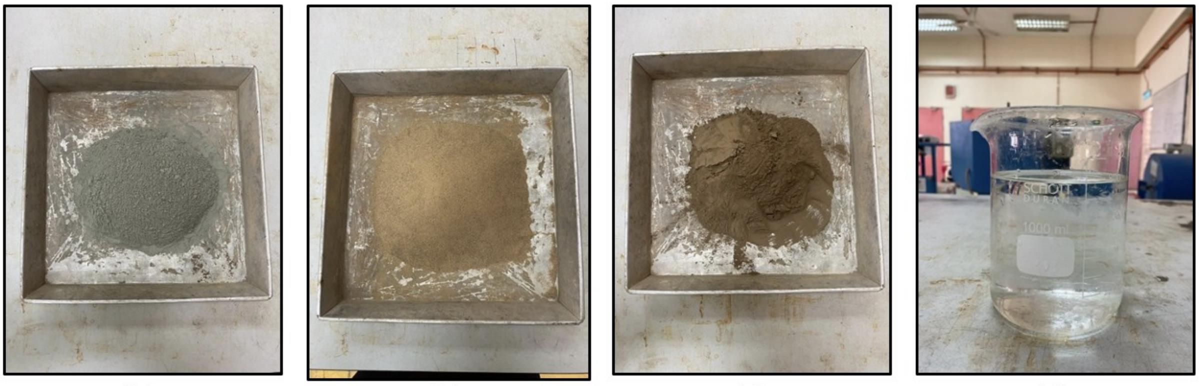

(a) (b) (c) (d)

Figure 1. The samples of (a) OPC, (b) river sand, (c) EPP, and (d) water.

2.2. Mixture Proportions of Mortar Samples

Table 1 shows the mixture proportion of mortars prepared according to ASTM C109 [21]. There were seven (7) specimens, including the control specimen and specimens containing EPP at 10%, 20%, 30%, 40%, 50%, and 60%. The water-to-cement (w/c) ratio used in this study was 0.485 for the control sample.

Table 1. Summary of specimen designation and mixture proportions

Specimen	Cement (g)	Sand (g)	EPP (g)
Control	1000	2750	0
E10	900	2750	100
E20	800	2750	200
E30	700	2750	300
E40	600	2750	400
E50	500	2750	500
E60	400	2750	600

2.3 Testing Procedures

2.3.1. Material Characterization

This study examined OPC and EPP's chemical, mineralogical, microstructural, and physical properties. The material's chemical composition, mineralogical composition, and microstructural properties were determined using X-ray fluorescence (XRF) and X-ray diffraction (XRD), respectively. The specific gravity of the materials was determined following ASTM C188 [22]. Meanwhile, the particle size analyser (PSA) was used to evaluate the particle sizes at d10, d50, and d90. All the characterisation tests for the materials were sent to the reference laboratories for testing, which are more specialised, as tabulated in Table 2.

Table 2. Summary of tests and laboratories

No	Properties	Tests	Laboratories
1	Chemical composition	XRF	Slag Cement Sdn. Bhd.
2	Mineralogical composition	XRD	Slag Cement Sdn. Bhd.
3	Microstructural properties	SEM	Biotechnology Research Institute (BRI), UMS
4	Specific Gravity	ASTM C188	Geospec Sdn. Bhd.
5	Particle size distribution	PSA	Geospec Sdn. Bhd.

2.3.2. Mortar Sample Preparation and Tests

The mixing procedure was conducted using a mechanical mixer, following the guidelines outlined in ASTM C305 [23]. After mixing, the fresh mortar was poured into a 50 x 50 x 50 mm mould and allowed to cure for 24 hours. Subsequently, the specimens were unmoulded and stored in water for 7 and 28 days. This was done to ensure the specimen attained uniform saturation, minimised leaching, and matured uniformly.

The flowability of the fresh mixtures was measured according to ASTM C1437 [24]. For mortar containing EPP, the water was adjusted to obtain a flow ± 5 of the value of the control mortar, as specified in ASTM C311 [25]. The water required to acquire the flow as per the standard was recorded. Then, the compressive strength of the specimens was measured according to ASTM C109 [21]. Then, the strength activity index (SAI) of EPP at 7 and 28 days was determined according to ASTM C618 [26]. Fig. 2 portrays the instrumentation employed in the study.

(a) (b)

Figure 2. The (a) flowability and (b) compressive strength used to test the properties of the specimens.

3. RESULTS AND DISCUSSION

3.1. Chemical Properties

Table 3 tabulates the chemical oxides of OPC and EPP. EPP's calcium oxide (CaO) content was significantly lower than OPC's, which aligns with past studies [5-11]. The results also reveal that the EPP had higher SiO_2 content than the OPC, with 51.78%, which falls within the range of previous studies [5-11]. Meanwhile, the sum of SiO_2, Alumina (Al_2O_3), and Iron Oxide (Fe_2O_3) was 73.06%. On the other hand, EPP had a lower sulphur trioxide (SO3) content of 0.11%. The results also reveal that the EPP's loss of ignition (LOI) was slightly higher than that of OPC at 3.26%.

From the chemical oxides results, the EPP is classified as Class N pozzolan as per ASTM C618 [26], which is consistent with findings by Lim et al. [6] and Rokiah et al. [7]. Other studies found that EPP was classified as Class C and F pozzolan [5, 9,10]. These differences may be due to the repetitive use of bentonite clay and the type of clay used in the degumming process, which can produce different concentrations of spent bleaching, ultimately making differences in the pozzolan classification of EPP [27].

Table 3. Chemical oxides of OPC and EPP

Chemical Compounds (%)	OPC	EPP	Class N
CaO	62.97	8.47	
SiO_2	19.65	51.78	
Al_2O_3	4.81	10.84	
Fe_2O_3	3.08	10.44	
$SiO_2 + Al_2O_3 + Fe_2O_3$	27.54	73.06	≥ 70.0
MgO	1.75	4.89	
SO_3	2.42	0.11	≤ 4.0
K_2O	0.64	1.4	
Na_2O	0.1	0.2	
P_2O_5	0.13	3.79	
TiO_2	0.31	1.52	
Mn_2O_3	0.19	0.24	
LOI	3.18	3.26	≤ 10.0

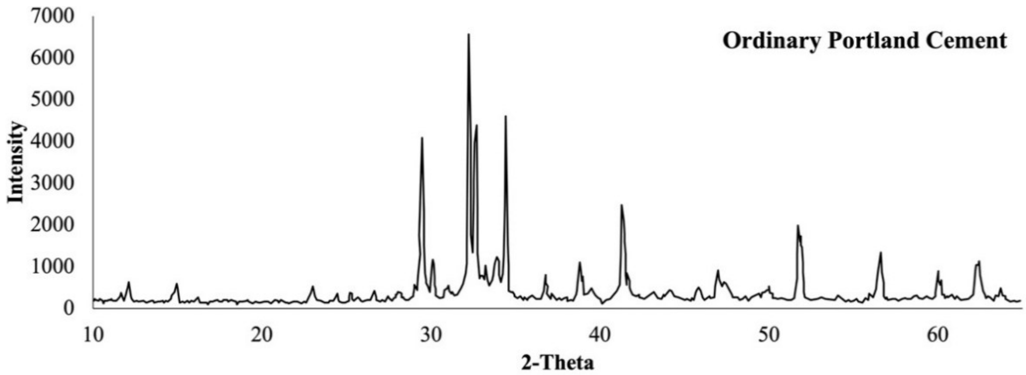
3.2. Mineralogical Properties

The OPC and EPP XRD analysis is tabulated in Table 4 and Fig. 3, respectively. The OPC comprised a high amount of Alite and Belite, with 65.65% and 10.26%, respectively. These compounds are responsible for developing the early and later strength of concrete. There were also traces of Ferrite (11.33%), Aluminate (3.87%), Calcite (3.51%), Hemi-hydrate (1.81%), and Portlandite (1.16%). Less than 1% of compounds include free lime (fCaO), Periclase, Gypsum, Anhydrite, Langbeinite, Aphthitalite, Arcanite, Lime, Quartz, and Dolomite.

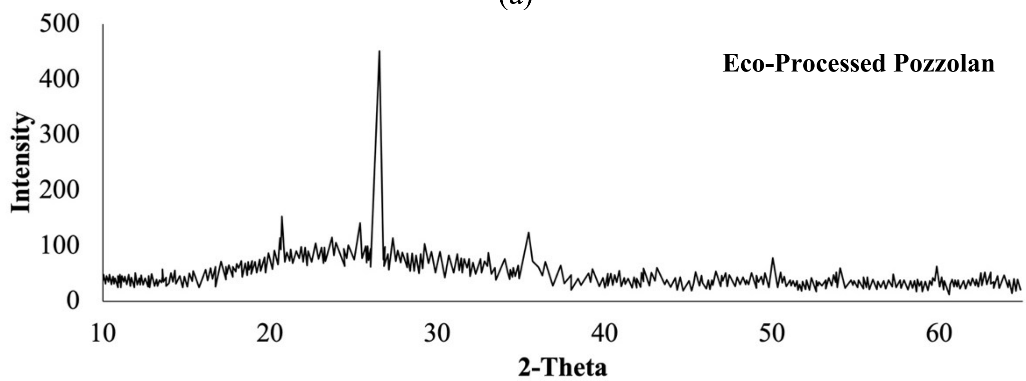
Meanwhile, EPP comprised high pozzolan amorphous silica with 64.28%, indicating pozzolanic reactivity [28]. The second-highest compound in EPP was Quartz, with 15.38%, which may contribute to pozzolanic reactivity [29]. Wafa Mechti found that the pozzolanic reactivity of crystallised quartz with fineness 20um at 10 % Portland cement replacement improved the cement paste compactness by increasing compressive strength and hydration [29]. Less than 10% compounds include Labradorite, Hematite, Magnetite, Calcite, Dolomite, Diopside, Olivine, Hornblende, Cristobalite, and Tridymite.

Table 4: Mineralogical properties of OPC and EPP

Compounds	OPC (%)	EPP (%)
Alite	65.65	-
Belite	10.26	-
Ferrite	11.33	-
Aluminate	3.87	-
Hemi-hydrate	1.81	-
Portlandite	1.16	-
fCaO	0.91	-
Periclase	0.76	-
Gypsum	0.41	-
Anhydrite	0.20	-
Langbeinite	0.18	-
Aphthitalite	0.10	-
Arcanite	0.07	-
Lime	0.03	-
Calcite	3.51	3.24
Quartz	0.43	15.38
Dolomite	0.22	2.27
Pozzolan amorphous Silica	-	64.28
Labradorite	-	6.07
Hematite	-	4.93
Magnetite	-	3.29
Diopside	-	0.08
Olivine	-	0.40
Hornblende	-	0.03
Cristobalite	-	0.02
Tridymite	-	0.01



(a)



(b)

Figure 3. XRD graph analysis of (a) OPC and (b) EPP

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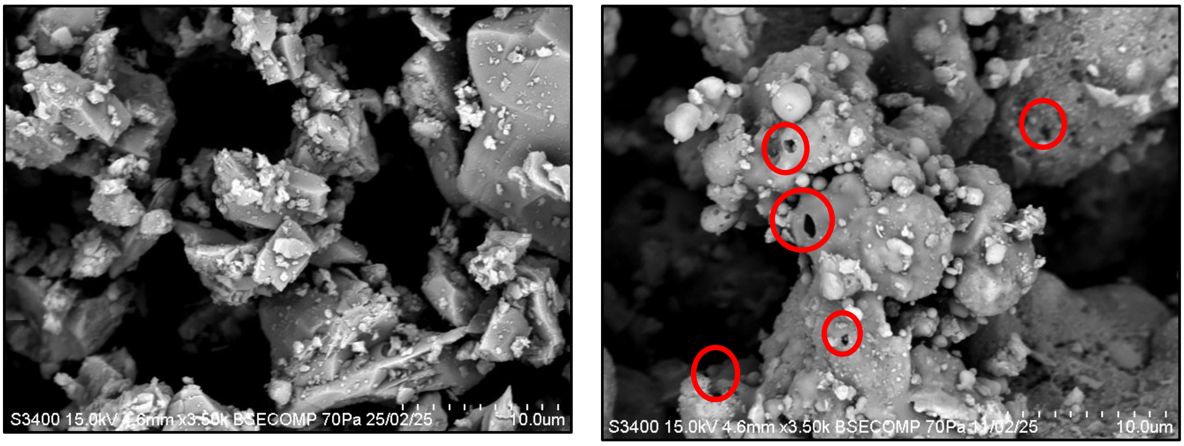
3.3. Microstructural Properties

Fig. 4 presents the SEM images of OPC and EPP, illustrating their respective morphologies. The image reveals that the OPC particles had an angular and irregular structure, consistent with past studies [5,7]. This means that they do not have a uniform and consistent shape. The angular microstructure of the OPC may increase the surface area, which promotes bonding with constituent materials, thus resulting in better strength development [30]. Meanwhile, EPP mainly consists of spherical and porous structures (as indicated in red circles in Fig. 4(b)), with a minor presence of irregular particle shapes, which also aligns with previous studies [5-7,9,10]. The porous microstructure of EPP may inhibit the strength development of the mortar by allowing water to occupy the empty spaces; hence, as it evaporates, it leaves voids that decrease the mortar's strength [31].



(a) (b)

Figure 4. SEM images of (a) OPC and (b) EPP

3.4. Particle Size Distribution

Table 5 represents the physical properties of OPC and EPP. Based on the mean particle size in the table, EPP generally had a larger particle size than OPC. This observation is consistent with Lim et al. [6], who indicated that the EPP was typically coarser than OPC particles. The retained 45-micron sieve showed that EPP retained a smaller percentage than OPC. Coarser particles of EPP in mortar can reduce its compacting density as larger particles of EPP leave larger voids between particles, hindering the close packing of mortar. Lin et al. (2024) found that the increment of EPP replacement on cement reduced the ultrasonic pulse velocity test result, indicating a significant void in the matrix [9].

Based on the results, the specific gravity of OPC and EPP was 3.12 and 1.77, respectively. This indicates that EPP was lighter than OPC, which may be due to its porous structure, as mentioned in Section 3.3.

Table 5. Physical properties of OPC and EPP

Properties	Amount retained 45 μm sieve (%)	Mean particle size d10 (µm)	d50 (µm)	d90 (µm)	Specific gravity
OPC	0.88	0.070	0.162	0.261	3.12
EPP	0.94	0.230	0.354	0.493	1.77

3.5. Flow Spread and Water Requirement of Specimen

The specimen's flow spread and water requirement are tabulated and illustrated in Table 6 and Fig. 5, respectively. The amount of water was adjusted for the mortar with EPP to obtain ±5% of the control mortar as specified in ASTM C311. Based on the result, the water requirement increased with the EPP replacement level. This indicates that high EPP content caused more water to be absorbed by the particles due to their porous texture, as stated in Section 3.3. Moreover, EPP also had a lower specific gravity, which may contribute to the increased demand for water. This finding is consistent with Kho [12], who stated that the finer size and higher porosity of EPP require a higher amount of water, thus causing a slump reduction of concrete.

Table 6. Flow and water requirement of the specimen

Specimen	Flow Spread (mm)	Water Requirement (%)
Control	223	100
E10	223	102
E20	223	106
E30	225	107
E40	223	111
E50	225	114
E60	223	125

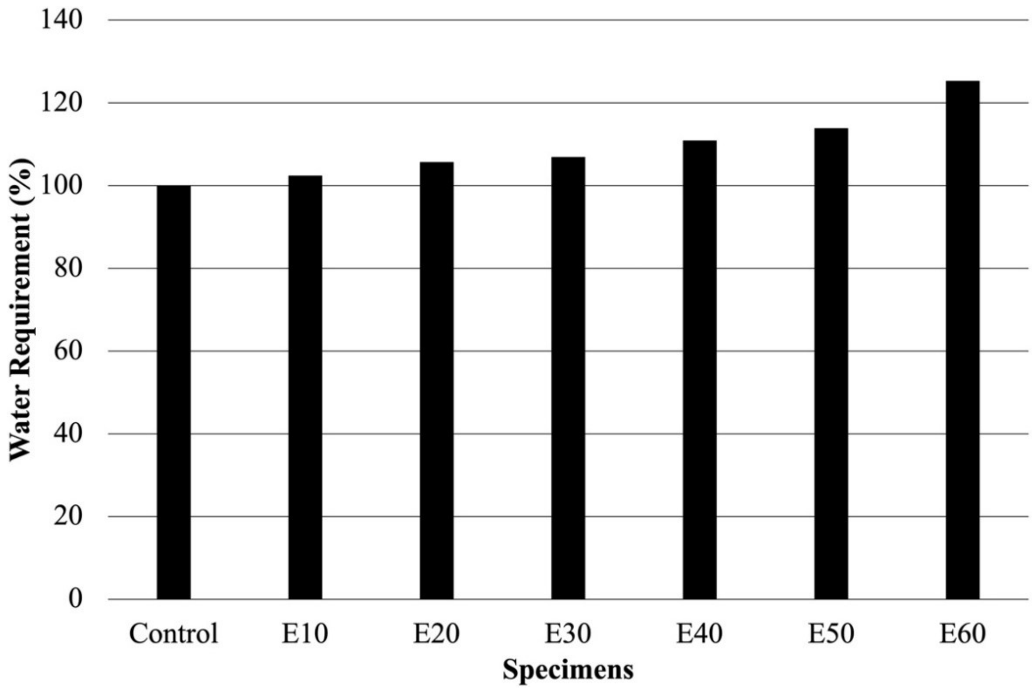


Figure 5. Water requirement of specimens

3.6. Compressive Strength and Strength Activity Index

Table 7 presents the compressive strength of mortar at 7 and 28 days. At 7 days, E10 exhibited the highest compressive strength of 19.2 MPa, which was 13.6% higher than that of the control specimen. Then, there is a decline in strength from E20 to E60, with strength lower than the control by a 3.6% to 75.7% reduction. At 28 days, the compressive strength of E10 and E20 was 14.3% and 4.9% higher than that of the control specimen. However, E30 to E60 had lower compressive strength than the control, declining from 2.7% to 67.6%.

The SAI of the specimens at 7 and 28 days are illustrated in Fig. 5. At 7 days, the SAI values of E10, E20, and E30 were 113.6%, 96.4%, and 78.% respectively, which were more than the minimum requirement of 75% SAI value as stipulated in ASTM C618 [26] (as shown in blue line in Fig. 6). Meanwhile, E40, E50, and E60 did not achieve the minimum requirement. This is because the amount of CaO becomes lower as the amount of EPP increases, which leads to low formation of calcium hydroxide (Ca(OH)2) [5,32,33].

At 28 days, the SAI values of E10, E20, E30, and E40 achieved more than the minimum requirement as per ASTM C618 [26], with 114.3%, 104.9%, 97.3%, and 84.6%, respectively. However, the remaining specimens did not achieve the minimum requirement. According to Abd Rahman et al. [5], the SAI value increment at 7 days and 28 days was due to higher SiO2 content, which enhances the pozzolanic reaction with Ca(OH)2 that occurs at a later stage of strength development [7]. This led to more CSH gel production, thus resulting in high compressive strength [5-7]. Among all the specimens, E10 and E20 had higher SAI values than the control specimens, indicating that 10% and 20% EPP are the optimum levels for cement replacement.

Table 7. Compressive strength and SAI

Specimen	Compressive Strength		SAI (%)	
	7 Days	28 Days	7 Days	28 Days
Control	16.9	18.2	100	100
E10	19.2	20.8	113.6	114.3
E20	16.3	19.1	96.4	104.9
E30	13.2	17.7	78.1	97.3
E40	10.2	15.4	60.4	84.6
E50	7.9	11.2	46.7	61.5
E60	4.1	5.9	24.2	32.4

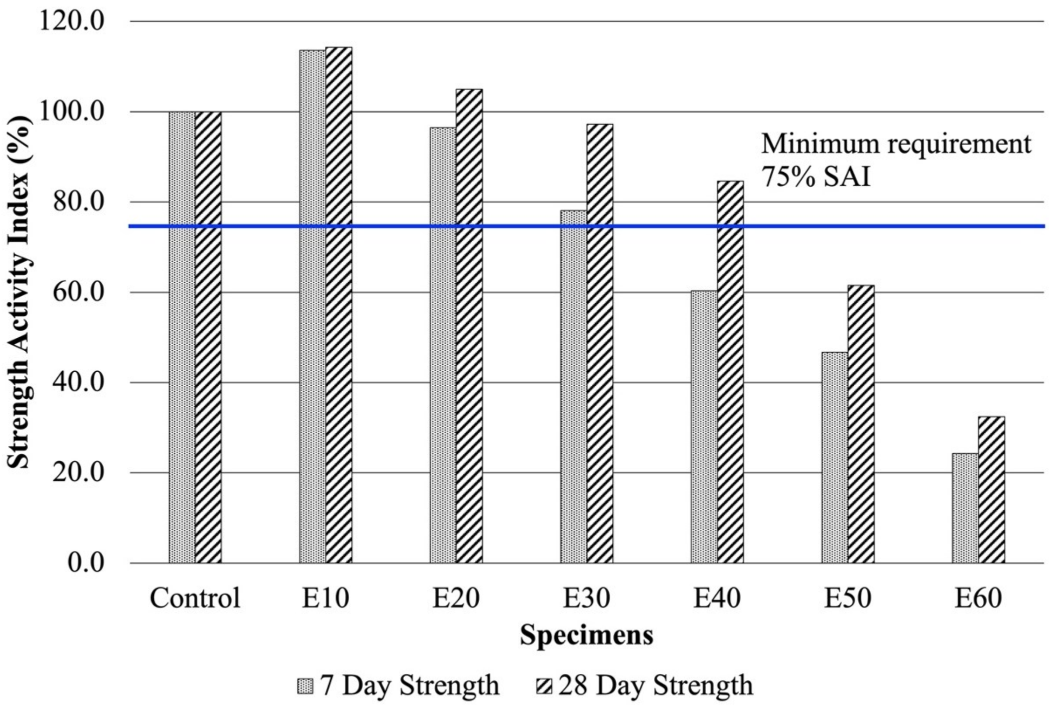


Figure 6. Strength Activity Index (SAI) at 7 and 28 days

In summary, the data obtained from this study may be influenced by environmental variation or experimental error, which could introduce inconsistencies in the conditions under which the samples were prepared, cured, or tested. Factors such as fluctuations in temperature and humidity, variations in mixing techniques, or inconsistencies in the quality of raw materials, especially the eco-processed pozzolan mineralogical and chemical properties, could lead to differences in water demand, water/ binder ratio, or strength development. Additionally, human error during the weighing, mixing, or testing process may have contributed to deviations in the data. These uncontrolled variables can impact the reliability and accuracy of the results, making it difficult to draw definitive conclusions about the actual performance of the pozzolan-blended concrete.

4. CONCLUSION

EPP fulfilled the criteria as a Class N pozzolan as per ASTM C618 and thus can be used as a replacement for cement. The results revealed that the sum of SiO_2, Al_2O_3, and Fe_2O_3 was 73.06 (≥ 70%), the amount of SO_3 was 0.11% (≤ 4%), and the amount of LOI was 3.26% (≤ 10%). The high porosity of EPP particles increased water absorption, which could negatively impact the strength of the mortar.

It was also found that the SAI was reduced with high EPP replacement percentages due to lower CaO content. Meanwhile, the SAI values satisfied the minimum requirement of pozzolan as per ASTM C618 for EPP at cement replacement of 10, 20, 30, and 40%. However, the strength of the mortar was reduced below that of the control specimen when the EPP replacement was up to 30%. Lastly, EPP performed well as a cementitious supplementary material, with 10% and 20% as an optimal cement replacement.

The results have shown that EPP can be used as a partial cement replacement in concrete. This can reduce the reliance on traditional cement in concrete production, which has a higher carbon footprint. Meanwhile, it can also reduce raw material costs and energy consumption in cement manufacturing, ultimately decreasing the overall cost of concrete production. Furthermore, the improved performance of concrete with EPP as partial cement replacement can lead to longer-lasting structures and reduced maintenance needs.

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