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PHYSICAL AND MECHANICAL PROPERTIES OF GREEN CEMENTLESS MORTAR INCORPORATING WASTE PAPER SLUDGE ASH

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ABSTRACT: As an alternative to conventional construction material such as normal concrete, waste paper sludge ash (WPSA) based geopolymer is seen as a promising and viable option in construction material selection due to its high amount of aluminum (Al) and silicon (Si) content. This research aims to determine the microstructure and mechanical characteristics of WPSA in geopolymers. The alkaline solution that contains 6 M of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) was used to activate the geopolymer. The hardened 50 mm-sized mortars were prepared and underwent a heat-cured process for 1 day at various temperatures at 24 °C, 60 °C, and 90 °C, respectively. Then, the mortar cubes were placed in the laboratory until the testing days. A compression test was conducted to identify the strength development of the WPSA-based geopolymer mortar at 7, 14, and 28 days, respectively. Chemical composition was analyzed using X-ray fluorescence (XRF). Furthermore, Fourier-transformed infrared spectroscopy (FTIR) was conducted to ascertain the structural elucidation and scanning electron microscope (SEM) analysis was done to provide microstructural observations of the geopolymer. Based on the XRF analysis, the WPSA has the highest amount of calcium oxide (CaO) instead of aluminum oxide (Al_2O_3) and silicon dioxide (SiO_2), and it reduces the performance of WPSA as a cement replacement material. The ratio of SiO_2 and Al_2O_3 is recorded as 1.1:1. Therefore, it is suitable for bricks and ceramics production instead of concrete production. As for the curing process, the heat-cured method is evident in accelerating strength development in the WPSA-based geopolymer mortar compared to the ambient curing method due to the rapid polymerization process in the geopolymer system. It is proven that 60 °C is the optimum temperature for the curing process for geopolymer mortar.

ABSTRAK: Sebagai salah satu alternatif kepada bahan binaan konvensional seperti konkrit biasa, geopolimer berasaskan abu enap cemar kertas (WPSA) adalah dilihat sebagai pilihan yang baik kerana bahan ini mempunyai kandungan aluminium (Al) and silika (Si) yang tinggi. Penyelidikan ini bertujuan untuk menentukan struktur mikro dan ciri mekanikal WPSA dalam geopolimer. Larutan alkali yang mengandungi 6 M natrium hidroksida (NaOH) dan natrium silikat (Na_2SiO_3) digunakan untuk mengaktifkan geopolimer. Mortar yang bersaiz 50 mm telah disediakn dan ia melalui proses pengawetan haba selama 1 hari pada suhu yang berza-beza, iaitu 24 °C, 60 °C, and 90 °C. Kemudian, kiub mortar tersebut diletakkan di dalam makmal sehingga hari ujian. Ujian mampatan dijalankan untuk mengenal pasti perkembangan kekuatan mortar geopolimer apabila mencapai 7, 14, dan 28 hari. Komposisi kimia dalam sampel telah dianalisa menggunakan pendarfluor sinar-X (XRF). Tambahan pula, Spektroskopi inframerah fourier transformasi (FTIR) untuk memastikan sifat bahan dan analisis pengimbasan mikroskop elektron (SEM) untuk menyediakan pemerhatian struktur mikro geopolimer. Berdasarkan data dari analisa XRF, WPSA mempunyai jumlah kalsium oksida (CaO) tertinggi berbanding aluminium oksida (Al_2O_3) dan silikon dioksida (SiO_2), dan ia mengurangkan prestasi WPSA sebagai bahan gantian simen. Nisbah SiO_2 dan Al_2O_3 direkodkan sebagai 1.1:1. Oleh itu, ia sesuai digunakan untuk pengeluaran batu bata dan seramik berbanding pengeluaran konkrit. Bagi proses pengawetan, kaedah pengawetan haba terbukti mempercepat perkembangan kekuatan geopolimer berasaskan WPSA berbanding pengawetan pada suhu ambien disebabkan berlakunya proses pempolimeran yang sangat pantas dalam sistem geopolimer tersebut. Telah terbukti bahawa suhu 60 ℃ adalah suhu optimum bagi proses pengawetan mortar geopolimer berasaskan WPSA ini.

KEYWORDS: *Geopolymer, Waste Paper Sludge Ash, NaOH,* Na_2SiO_3.

1. INTRODUCTION

Nowadays, ordinary Portland cement (OPC) is widely used as a construction material worldwide [1–3]. Cement production is one of the largest sources of CO2 emitted into the atmosphere than deforestation and fossil fuels [4], [5]. The high demand for cement production is rising rapidly day by day because of the increased need for building construction materials. Cement manufacturing is the source of harmful gas outflow to the earth's atmosphere, which is assessed at around 1.5 billion tons every year [3]. However, the expected value is greater than 4.8 billion tons of CO2 production in 2020 [6].

On the other hand, waste materials generated from rapid industrial activities continue to expand each day [7]. Managing the disposal of these waste materials requires proper landfilling techniques. The amount of waste generation per capita in Malaysia is about 1.1kg/day [8]. The per capita generation of municipal waste is from 0.5 to 0.8 kg/ person/ day [7]. In addition, 166 landfills have been operated to dump more than 26,500 tons of waste every day in the country [8]. Among the commonly generated waste is paper waste. This type of waste is the most frequently found in almost all areas. The paper can only be recycled six to ten times because of the decreasing strength of the cellulose fiber of the paper. After the recycling process, the rest of the waste paper will be dumped into landfills. Paper production industries contribute a significant amount of waste materials since the material can only be recycled a few times, which is before its fiber becomes weak to produce the high-quality end product such as paper tissue [9]. Therefore, exploring the potential to use this waste material in the construction industry is seen as one green solution to address the issue accordingly.

In this regard, geopolymer is an environment-friendly construction material that utilizes industrial or agricultural waste materials mixed with an alkaline activator as the binder to bind sand instead of OPC [10]. Examples of waste materials used in geopolymer are fly ash, slag, silica fume, bagasse ash, clay, rice husk ash, and metakaolin [11], [12]. Waste paper sludge ash (WPSA) that consists of high amount of alumina-siliceous material can be a possible cement substitution in the construction industry [13], [14]. The alumina-siliceous is crucial in geopolymer technology, as it enhances good physical and chemical characteristics. SiO_2 and Al_2O_3 presented in the WPSA react with an alkaline-activated solution to produce an excellent geopolymer with a high alumina-siliceous material [15]. This reaction is known as the geopolymerization process, involves the dissolution of silicate and aluminate substances and polycondensation reaction [6]. The end product of this process is silicon oxo aluminates, which look like a three-dimensional (3D) polymeric ring structure. Thus, the WPSA enhances the performance of geopolymer [16], [17]. Contrasting to the OPC, the binder is calcium-silicate-hydrates to develop strength [18]. A geopolymer required an aluminosilicate to cohere the aggregates and enhance the strength [16].

Using geopolymer innovation is one of the feasible strategies to lessen the utilization of OPC in concrete, which causes environmental contamination and expands the measurement of waste materials. Therefore, the best way to ensure that the concrete industry is more competitive is by using waste and natural materials. Geopolymer is a new green concrete concept that uses eco-friendly materials in cement production. Geopolymer is an excellent substitute for cement to reduce pollution. Geopolymer cement can contribute considerably to the carbon dioxide discharges in the atmosphere compared to OPC. In addition, many researchers [9], [14], [20], [21] have stated that WPSA can be used as cement replacement material as well. Therefore, an effort has been made to optimize the usage of recycled waste materials as beneficial alternative materials for cement [17]. The geopolymer is the best way to replace OPC in concrete.

In this regard, this research used WPSA as the main constituent in the geopolymer. Thus, the research aimed to find the mechanical properties through a compressive strength test and microstructure properties through X-ray fluorescence (XRF) analysis, scanning electron microscopy (SEM) analysis, and Fourier-transformed infrared spectroscopy (FTIR) analysis of WPSA-based geopolymer.

2. MATERIALS AND METHODS

2.1. Material Selection

2.1.1. Waste Paper Sludge Ash (WPSA)

As shown in Fig. 1, WPSA is waste material directly taken from the local paper milling factory, Malaysian Newsprint Sdn Bhd, located in Mentakab, Pahang. The WPSA is light grey and a by-product of the paper milling process. The percentage of WPSA used as a binder in geopolymer mortar is 100% to replace cement.

Figure 1. Waste Paper Sludge Ash

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2.1.2. Fine Aggregates

For this study, a sieve analysis test was done to determine the grain size distribution of fine aggregate particles (sand). Commonly, the grading of fine aggregates is defined as the proportions of particle size less than 5 mm. The sieve analysis produced a grading curve for fine aggregate according to ASTM C136/C136-19. The grading curve is plotted in the percent passing (%) graph versus sieve size (mm), also known as the particle size distribution graph for fine aggregates.

Gradation and size distribution of the fine aggregates affect the mechanical and microstructure properties of the produced geopolymer mortar. Commonly, well-graded aggregates contribute towards compressive strength increment for the mortar. From the data in Table 1, the total weight of the fine aggregate was 5200.00 g, while the total weight was calculated at 5004.20 g. Thus, there was a 195.80 g loss of fine aggregates. Therefore, some errors might affect the experiment's accuracy, such as the fine aggregates that have been used may contain unwanted materials such as dust and stone.

Table 1. Sieving analysis of aggregates

Sieve size (mm)	Weight of sieve (g)	Weight of sieve + sample (g)	Weight of sample (g)	Cumulative weight sample (g)	Cumulative % retained (%)	% passing (%)
5.00	0.00	0.00	0.00	0.00	0.00	100.00
2.36	503.20	754.30	251.10	251.10	5.02	94.98
1.18	704.40	1390.00	685.60	936.70	18.72	76.26
0.60	903.80	2206.30	1302.50	2239.20	44.75	31.52
0.30	908.90	2610.00	1701.10	3940.30	78.74	24.00
0.15	1223.90	1931.40	707.50	4647.80	92.88	10.00
pan	748.10	1104.50	356.40	5004.20	100.00	0.00

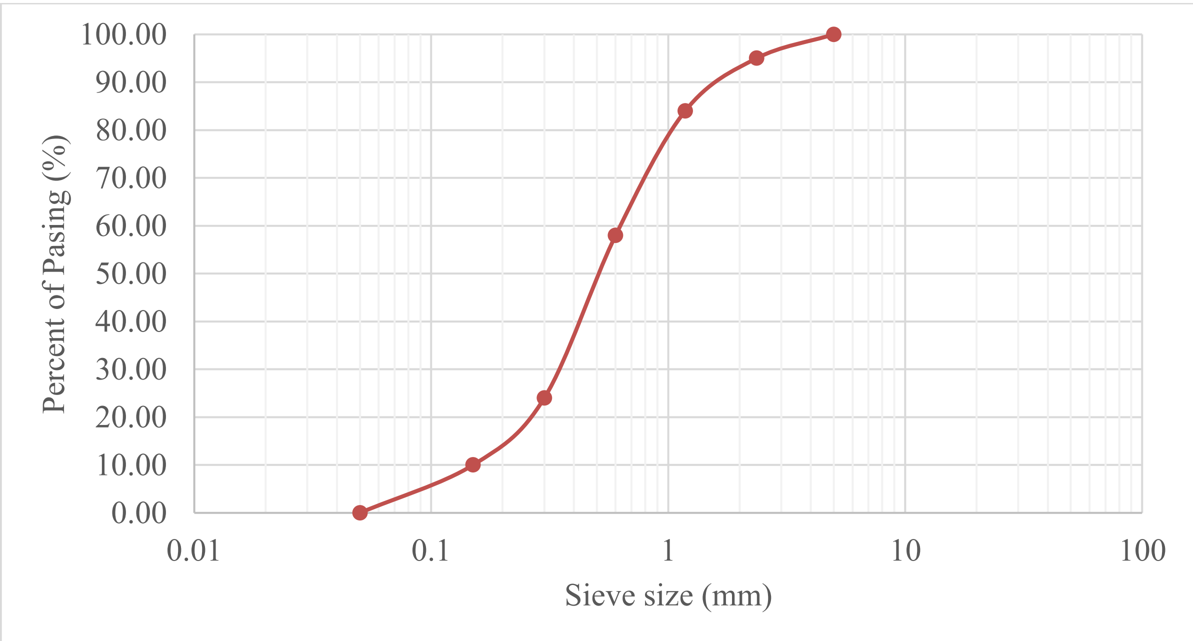

Figure 2. Particle size distribution of fine aggregates.

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Fig. 2 shows the grading curve of the particle distribution of sand for the geopolymer mortar obtained from sieve analysis. According to the standard ASTM C136/C136-19, it is a well-graded aggregate containing sand particles retained on each sieve stack that is less than 5 mm. Steven et al. [22] Describe how well-graded aggregates give great workability and durability. The gap between aggregate particles that paste must fill is reduced when they are well-graded aggregates. A good bond between WPSA and sand is created since the sand can be compacted to reduce the geopolymer mortar's air void and porosity.

2.1.3. Alkaline Solution

An alkaline solution is a combination of (sodium hydroxide) NaOH solution and (sodium silicate) Na_2SiO_3 solution. 6 M of NaOH solution was used in this research. Na_2SiO_3 solution combined with NaOH solution to form an alkaline solution. The chemical composition of the Na_2SiO_3 solution adopted was Na_2O = 16.5 %, SiO_2 = 50.60%, and water = 32.90% by mass, respectively. The density of Na_2SiO_3 is 1.350-1.380 units.

2.2. Mix Proportions

30 samples of geopolymer mortars were prepared in this research. The ratio of WPSA to sand (W/S) is 1: 2.75 based on ASTM C109/C109M. (W/S) adopted was constant for all samples. 6 M of NaOH solution and Na_2SiO_3 activated the geopolymer mortar. The ratio of Na_2SiO_3 and NaOH was fixed and applied as 4:1 to all samples to produce an alkali activator, as reported by other researchers as well [20]. The ratio is considered an optimum value for producing high aluminosilicate in geopolymer to bind aggregates and develop strength. NaOH is less than Na_2SiO_3 because enormous NaOH can disturb the end product properties. However, an adequate amount of Na_2SiO_3 ensures sufficient silica is available for network formation. Adding Na_2SiO_3 solution to NaOH solution significantly enhances the reaction between the waste material and the alkaline solution [23]. NaOH diffuses aluminosilicate material, while Na_2SiO_3 helps NaOH act as a binder in the mortar composition [24]The ratio of water to material (W/M) is 0.25. The material is a combination of sand and WPSA.

To prepare WPSA-based geopolymer mortar samples, a similar procedure of conventional cement-based mortar production in accordance with ASTM C451 is implemented. The aim is to observe the effect of various curing temperatures on the properties of geopolymer containing WPSA and the compressive strength conducted on the hardened specimens. Table 1 shows the proportions of the geopolymer mixture. From Table 2, the polymeric mortar mixtures were mixed with the same amount of WPSA and sand.

Table 2. The proportion of the mixture of geopolymer (kg/m^3)

Mix Code	WPSA	Sand	Alkaline solution/ material ratio (L/M)	NaOH	Na2SiO3	Total water/materials ratio (W/M)
WPSA-T24	300	825	0.6	135	540	0.25
WPSA-T60	300	825	0.6	135	540	0.25
WPSA-T90	300	825	0.6	135	540	0.25

2.3. Curing Method

This study determines the effect of WPSA-based geopolymer at three different curing temperatures. Table 3 shows various curing temperatures for the specimens. The curing method is the most important process that has to be properly conducted to develop the strength of the specimens. The size of the test specimens was 50 mm x 50 mm x 50 mm, and they went through heat-cured in an oven at various temperatures for 1 day. Cubes that underwent a heat-cured process were placed in a drying oven, as shown in Fig. 3. After the curing process, the specimens were left for about 1 hour to cool down and then placed in the laboratory until the testing day.

Table 3. Curing temperature of geopolymer mortar

Mix Code	No of samples	Curing temperature (°C)
WPSA-T24	10	24
WPSA-T60	10	60
WPSA-T90	10	90

Figure 3. Heat-cured process.

2.4. Geopolymer Testing Method

2.4.1. Characterization Properties Test

For this research, three analyses were conducted for the physical properties test of WPSA. Firstly, the X-ray fluorescence (XRF) Analysis is used to determine the chemical composition of geopolymer mortar. XRF analysis defines the arrangement and types of chemical substances in the geopolymer. This research used a 5-gram WPSA powder sample. Next, the morphologies of WPSA images are shown through Scanning Electron Microscopy (SEM) analysis according to ASTM C1723-16. The magnifications of SEM images of WPSA are at 170 and 500 times. This study used 5 grams of WPSA to undergo this test. Lastly, the geopolymer pastes were prepared for Fourier Transformed Infrared Spectroscopy (FTIR) analysis. The FTIR analysis conducted for the age of geopolymer pastes reached 7 and 28 days based on ASTM E168.

2.4.2. Compressive Strength Test

A compressive strength test was conducted to measure the hardened mortar's compressive strength development and analyze the impact of WSPA-based geopolymer. After the 50 mm ×

50 mm × 50 mm cubes had aged 7, 14, and 28 days, the compressive strength of each geopolymer mortar was measured according to ASTM C109/C109M.

3. RESULT AND DISCUSSION

3.1. Characterization Properties of Waste Paper Sludge Ash (WPSA)

3.1.1. Chemical Composition and Morphology of Waste Paper Sludge Ash (WPSA)

The chemical composition of WPSA was determined through XRF analysis and tabulated in Table 4. From the XRF observation, the WPSA contains calcium oxide (CaO) as a major composition. WPSA as a cement replacement material depends on the ratio of Si: Al. The ratio of SiO_2 and Al_2O_3 is 1.1:1 and is unfavorable for geopolymer use. Newman et al. [17] stated that the ratio of Si: Al is 1.1:1, which is suitable for producing bricks and ceramics. Many past researchers stated that the ratio of SiO_2 and Al_2O_3 should be 2:1 in geopolymer [13], [15]. The presence of high CaO in geopolymer affected the cations, caused a rapid stiffening, and altered the output of the polymerization [20]. Thus, Rahman et al. [20] concluded that the high amount of CaO in WPSA could disturb the polymerization process, causing changes in the microstructure of geopolymer mortar. This is because a geopolymer is not similar to normal concrete with 100% OPC, which requires the calcium-silicate hydrates to be involved in the polymerization process to develop strength [14]. Despite the low presence of SiO_2 and Al_2O_3 in WPSA, further mechanical tests, namely compressive strength tests, need to be conducted to confirm the trend.

Table 4. Chemical composition of WPSA

Chemical oxide	SiO_2	MgO	Al_2O_3	P_2O_5	K_2O	Na_2O	Fe_2O_3	SO_3	TiO	CaO
Chemical content (%)	17.14	5.54	15.00	0.24	0.58	0.64	0.44	0.09	0.18	60.15

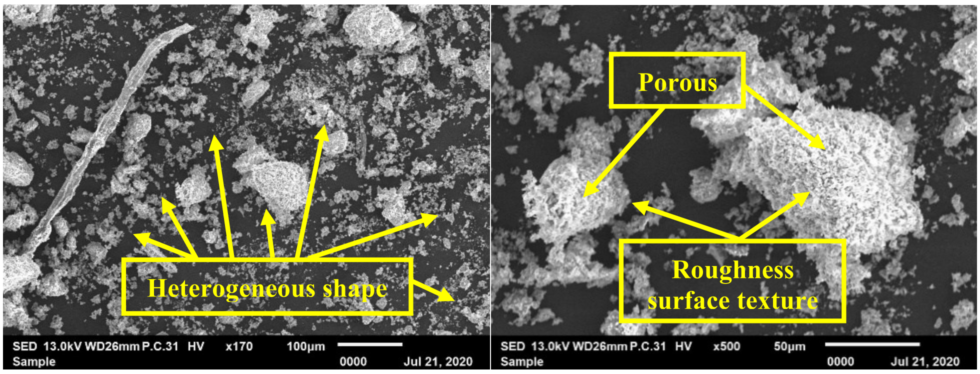


Figure 4. SEM images of WPSA, (a) Magnification 170 times,
(b) Magnification 500 times.

Fig. 4 (a) and Fig. 4 (b) are the SEM images of WPSA at magnification 170 and 500 times. The SEM images of WPSA showed a highly porous structure and agglomerated particles. So, it causes problems with the workability of geopolymer mortar and geopolymer paste. The same observations were made by past researchers regarding the WPSA powder [17–19] [20]–[22]. All the authors observed that the WPSA has porous and agglomerated particles. Mamat et al. [21] stated that the agglomeration of isolated mineral grains results from the burning process. SEM observations show that the WPSA particles do not have a consistent pattern and shape, such as irregular and non-uniform shapes. The pattern and shape inconsistency of WPSA showed a large range of particle sizes that created a good interlocking with aggregates and then enhanced the strength of geopolymer [25]. Fig. 3 (b) shows a close view of the rough surface texture of WPSA particles. The rough surface of WPSA particles created high friction between aggregates and produced strong bonds.

3.1.2. Structural Elucidation of Geopolymer

Then, the paste composition of a geopolymer undergoes the Fourier-transformed infrared spectroscopy (FTIR) at a wavenumber of 4000 – 500 cm^{-1}. This analysis aims to find the functional groups of the chemical compound in the WPSA-based geopolymer. The geopolymer pastes at the age of 7 and 28 days were used for this analysis.

Based on Fig. 5, the wave number of the O-H is recorded between 3500 and 4000cm-1. Other researchers similarly report this finding [13]. In addition, the Si-O-Si and Al-O-Si stretching vibrations were detected at the wavenumber 1200-950cm^{-1}. The degree of polymerization is related to Si-O-Si and Al-O-Si stretching [26]. This increase in the Si-O-Si and Al-O-Si stretching band increases the polymerization process. However, the increase of C-O stretching at the wavenumber of 1400 - 1450 cm^{-1} indicated the $CaCO_3$ because of the high calcite in WPSA [26].

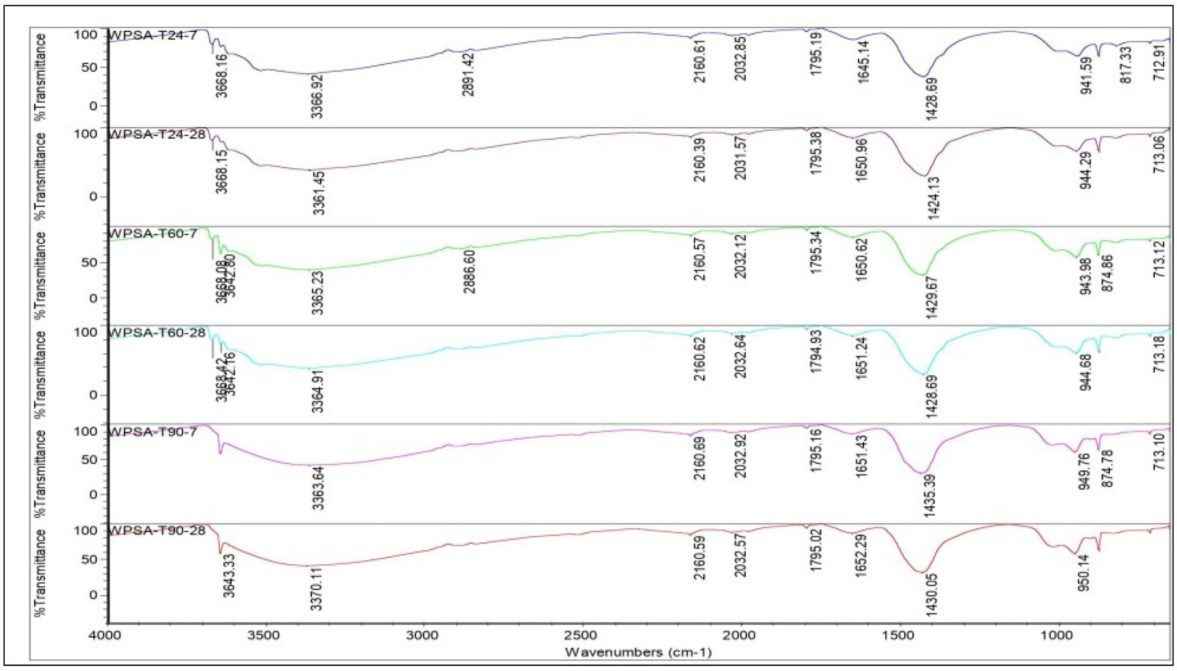


Figure 5. FTIR analysis for geopolymer pastes at 7 and 28 days.

3.2. Compressive Strength Results of Geopolymer Mortar

Based on Fig. 5, the geopolymer mortar cured at 60ºC has 4.65 MPa strength as the highest compressive strength at seven days. After that, the compressive strength of all geopolymer mortars shows an increasing pattern in the following seven days. For 14 days of compressive strength results, the geopolymer mortar cured at 60ºC still has the highest strength, 6.25 MPa. On the other hand, the geopolymer mortar samples cured at 24ºC gave the lowest strength, which is 4.24 MPa. Then, all geopolymer mortars' strength are expected to increase at 28 days based on the trend shown at 7 and 14 days, respectively. The geopolymer mortar cured at 60ºC still has the highest strength, 6.54 MPa, while the geopolymer mortar samples cured at 24ºC are the least.

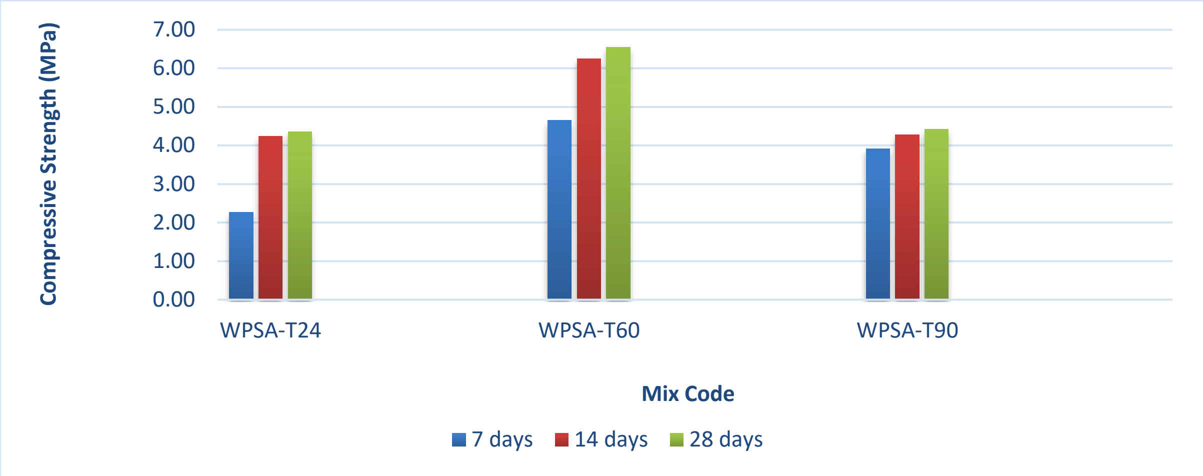
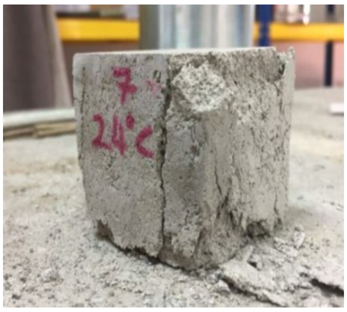


Figure 5. The comparison of compressive strength of mortar cubes at 7, 14, and 28 days.

Table 5. Observation of WPSA at 24ºC, 60ºC and 90ºC at 28 days

Specimens	**Compressive Strength Test**	
	Before	After
WPSA-T24		
WPSA-T60		
WPSA-T90		

In addition, Table 5 shows specimens cured at 24ºC, 60ºC, and 90ºC at 28 days before the compression test and after being set under the compressive load during the test until it reached failure. From the observation of images in Table 5, the geopolymer mortar cured at 60ºC had a few cracks appearing on the surface of the specimen after the test. Overall, the geopolymer mortar sample cured at 24 ºC has the lowest compressive strength for 7, 14, and 28 days of the curing process. Adam and Horianto [27] stated that heat-cured curing is more favorable than normal curing. The polymerization process occurred rapidly and developed more than 50% of the geopolymer mortar strength in the first four hours of heat curing. A geopolymer had a high development strength at an early cured period [28]. The geopolymer concrete cured at 24 ºC has poor development of compressive strength [29].

The findings show that the geopolymer mortar cured at 60 ºC has the highest strength throughout 28 days of the curing process. This finding agrees with Bhushan Jindal [29], who also summarized the optimum temperature and duration of the curing process for fly ash-based geopolymer suggested by past researchers, which is 60 ºC for 24 hours. In addition, Rajan and Ramujee [30] suggested that the optimum curing temperature is 60 °C for the size of the geopolymer mortar 50 mm cube. The 50 mm cube is small and cannot undergo a high curing temperature. The smaller size of the cube is more vulnerable and loses moisture content toward high curing temperature.

4. CONCLUSION

This research concludes that the Waste Paper Sludge Ash (WPSA) powder contains a higher amount of CaO compared to SiO_2 and Al_2O_3, which disrupts the polymerization process and slows the strength development of geopolymer mortar. The SiO2 to Al_2O_3 ratio of 1.1:1 is not ideal as a geopolymer mortar binder, where the optimal ratio for producing geopolymer is 2:1; however, the 1.1:1 ratio is suitable for brick and ceramic production. The preferred curing method for WPSA-based geopolymer mortar is heat curing, which significantly accelerates the polymerization process and strength development, with 60°C identified as the optimum temperature for curing. For future research, using WPSA as a sole binder is not recommended. Further studies should investigate the mechanical properties of the geopolymer mortar after 90 days of curing, as the strength would be fully developed by then. Additionally, conducting flexural and tensile tests is suggested to understand better the effects of these properties on the performance of geopolymer mortar.

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