INTERNATIONAL JOURNAL OF INTEGRATED ENGINEERING
ISSN: 2229-838X e-ISSN: 2600-7916

IJIE

Vol. 16 No. 8 (2024) 143-152
https://publisher.uthm.edu.my/ojs/index.php/ijie

Marble Waste Utilization in Geopolymerized Concrete Incorporating Outdoor Heat Exposure and Alkali Activated Material

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DOI: https://doi.org/10.30880/ijie.2024.16.08.015

Article Info

Received: 1 September 2024
Accepted: 5 November 2024
Available online: 13 Disember 2024

Keywords

Cementitious material, geopolymerization, alkali activated material, outdoor heat exposure, marble waste

Abstract

Marble waste can be incorporated in the geopolymerized concrete production as supplementary cementitious material. Therefore, this study intends to assess physical and mechanical properties of a geopolymerized product through marble waste utilization under outdoor heat exposure (OHE) and use of alkali activated material (AAM). In concrete production, marble waste was used at 20% by ratio and the specimen was subjected to OHE of 3-5 days and AAM/cement ratio of 0.3-0.4. Compressive strength analysis was performed to evaluate performance of geopolymerized concrete under the influence of OHE and AAM. The mineralogical and microstructure composition of the geopolymerized concrete were determined using XRD and SEM analysis, respectively. On average, specimens with 0.3-0.4 AAM/cement ratio and 3-5 days heat period have shown higher compressive strength than the control. The XRD and SEM analyses showed that the production of calcium silicate hydrate and other cementitious compounds were formed in the geopolymerized concrete product. Therefore, marble waste can be regarded as a suitable material to be used as supplementary cementitious material incorporating alkali activated material and outdoor heat exposure. This is in line with the concept of waste restoration in construction material for long-term environmental sustainability.

1. Introduction

The cement industry is already known as one of the industries with the most emissions toward the environment. Large scale activity of the cement industry takes up about 5% of greenhouse gases released into the atmosphere [1]. It can include carbon dioxide, sulfur dioxide, nitrogen oxide and other heavy-metal-based emissions [2]. Moreover, it is expected to increase as much as 27% by 2050 [1]. Prevention is needed to reduce the effect on the environment. Cement is highly requested due to its function as the main component for concrete is increasing due to the need for building and construction material. As the demand for cement is increasing, the cement industry also is getting busier, and cement will be produced on a large scale. Aside from producing cement, there are also other products released including the emission of greenhouse gases. The main factors of the cement industry that can cause detrimental effects toward the environment are the energy used, greenhouse gasses emission and solid waste. These factors will determine the degree of effects of the environment.

The mining sector has been producing a vast amount of waste [3]. This issue is very concerning and is detrimental towards the environment. Marble waste is one of the types of waste that has been produced by the mining sector. 20-30% of the marble work residue is produced as waste material [4]. Its production can degrade the environment. The production of marble waste in the form of fine marble dust can cause health problems, degrade soil quality and soil structure [4]. Therefore, action is needed to prevent the marble waste from degrading the environment by properly handling the waste. Marble waste is usually dumped into landfill because it does not benefit the mining industry.

However, it can be utilized into useful materials which can benefit the environment [5]. Marble waste can be utilized as supplementary cementitious material (SCM) for cement-based materials and is sustainable to the environment. The waste can be solidified into concrete and does not return back as waste to the environment. Solidification is a process of mixing a waste with a binding agent that can make all materials stick together. Then, the mixture is dried and forms a solid block. Solidification also can be applied for marble waste to produce concrete. Geopolymer concrete can also be made by utilizing marble waste. The geopolymerized concrete cured in high temperature can improve its strength development [6].

Geopolymerized concrete is a sustainable way of replacing the normal concrete. It can be fabricated by incorporating the three main components of geopolymer reaction such as alumino-silicate material, alkali activated material (AAM) and heat activation. Marble waste can be incorporated into the geopolymerized concrete production as the source of the alumino-silicate mineral. The alkali activated material can be used as precursor to enhance physical, mechanical and chemical properties of concrete including good strength performance [7]. While heat activation can be provided through the outdoor heat exposure (OHE). The OHE method can achieve up to 30°C to 40°C temperature through direct exposure to sunlight or heat source [8]. Many factors can influence the effects of OHE on geopolymerized products such as exposure time and use of alkali activation material. A larger degree of reaction can be found in longer OHE time [8].

As marble waste can be utilized in geopolymerized concrete production, the material can be reused to meet the cradle-to-cradle strategy. The goal of cradle-to-cradle strategy is eliminating waste by reusing or recycling waste material. Nearly 70% of the valuable mineral resources from the marble quarry and its production is wasted [9]. Thus, marble waste which contains valuable minerals can be recycled into useful material. It can be used as supplementary aggregates for cement-based materials which is one of the main components for geopolymer reaction. Furthermore, aggregates are needed for concrete material to have better strength, stability and durability [10]. Thus, marble waste is a potential candidate material that can be utilized in geopolymerized concrete production as supplementary cementitious material.

The aim of the study was to produce a green construction material which is of geopolymerized concrete with good physical and mechanical performance. It will also reduce energy consumption as it uses outdoor heat exposure as opposed to firing or oven heating while being able to maintain strength development. It is an attempt of this study to investigate the mineralogical and chemical properties of marble waste as supplementary cementitious material, assess the physical and mechanical properties of geopolymerized material under heat exposure and alkali-activated material, and evaluate the mineralogical and microstructure composition of the geopolymerized concrete products. Thus, aside from having mining waste securely managed, the geopolymerized concrete has the potential for revenue generation.

2. Materials And Methods

2.1 Sample Preparation

Samples of marble waste were ascertained from a marble production company located in Simpang Pulai, Perak. The samples were received as small fractions of between 1-10 mm in size. The marble waste was then crushed and grounded into fine sizes (<1mm) for subsequent analysis. The crushing process was handled using a cutter, hammer and crusher. The cutter was used to cut the marble waste into smaller sizes. Then, the material was crushed using a hammer and a crusher. In order to get a uniform size of marble waste (< 1 mm), a sieve was used to segregate the sizes. In the subsequent process for concrete making, the marble waste was used as a partial replacement for aggregates.

2.2 Outdoor Heat Exposure

The outdoor heat exposure (OHE) was provided through heating in a solar oven specifically designed to place the concrete cubes under natural exposure to sunlight. The solar oven was used to cure the geopolymer concrete replacing the microwave oven to minimize energy consumption by means of exposure to natural heating. It uses outdoor heat exposure to create a higher temperature inside the solar oven. Solar ovens are also environmentally-friendly as they use renewable energy to produce heat. By using the solar oven, greenhouse gasses emission can be prevented [11]. Box-type solar oven having a size of 60 cm x 60 cm was used with some modifications to achieve desired temperature (see Fig. 1). The solar oven keeps the temperature inside the oven by using solar energy. Specimens were placed inside the solar oven for heating and curing processes. For the purpose of the experiment, the solar oven was produced by using recyclable materials. The geometrical configuration of the solar oven played an important role in increasing the efficiency for heat trapping and maintaining the temperature during the curing process [12], [13].

Fig. 1 *Solar oven used for outdoor heat exposure*

2.3 Geopolymerized Concrete Production

The main components of the concrete production included marble waste, sand, cement and water. The marble waste concrete specimens were labelled accordingly. For samples B1, B2, B3 and B4, marble waste was used as partial replacement for aggregates at 20% replacement. The water:cement ratio, AAM:cement ratio and heat period were varied to observe the differences in the samples. A total of 3 control specimens were fabricated including normal concrete, use of heat and AAM (C1, C2 and C3). In the normal concrete, the cement:sand ratio was 1:4 with 0.5 water:cement ratio. For C2 and C3, the cement-sand-aggregate ratio was 1:1:3 with water:cement ratio of 0.5. The marble waste replacement in the specimens was kept at 20% as per regulated by Department of Public Works (JKR). The composition of the design mix for the concrete production is presented in Table 1. In this study, the concrete specimens were produced in cube sizes. The size of the cube was 50 mm x 50 mm x 50 mm. A total of 42 specimens were produced in this study for the analysis of concrete compressive strength, water absorption test and mineralogical analysis.

Table 1 *Design mix of concrete specimens*

Specimen	AAM[a]	Heat	Cement (%)	Sand (%)	Marble Waste[b] (%)	AAM: Cement Ratio[c]	Heat Period (days)
C1	N	N	20	80	-	-	-
C2	N	Y	20	60	20	-	5
C3	Y	N	20	60	20	0.4	-
B1	Y	Y	20	60	20	0.4	3
B2	Y	Y	20	60	20	0.4	5
B3	Y	Y	20	60	20	0.3	5
B4	Y	Y	20	60	20	0.3	3

[a] AAM – Alkali-activated material (NaOH)
[b] Marble waste composition is fixed at 20%
[c] Water:cement = 0.5.
Y/N (Yes/No) – with or without the presence of AAM/Heat

2.4 Compressive Strength Test

Compressive strength tests determine the load's carrying capacity [14]. As concrete will be used in buildings and construction areas, it is important to know how much a specimen of concrete can withstand the load. The compressive strength test was conducted by using a compression testing machine. The compression testing machine used a constant load with a uniform rate of 14 N/mm^2 until failure occurred. Snapshots of the specimen were taken to document the failure. The maximum load that can be taken by the concrete was the load at failure of the specimen. Compressive strength of the specimen was calculated using the formula:

$$\text{Compressive Strength} = \frac{\text{Maximum Load at Failure } (N)}{\text{Average Area of Bed Face } (mm^2)} \tag{1}$$

2.5 Water Absorption Test

The water absorption analysis can determine the water content absorbed in the concrete [14]. Water absorption is an important criterion for durable concrete [15]. The water will be absorbed into the concrete with different rates based on the mixing ratio. This analysis also can determine the percentage of pore volume of the concrete that is being filled in the period of soaking [14]. It is also known as the saturation coefficient. The specimen in cube shapes is dried using an oven. Then, the specimen will be put in a container filled with water and soaked for 24 hours. Water absorption can be evaluated through differences in weight before and after the specimen soaked into the water. The percentage, % of the water absorption can be easily calculated by using the formula:

$$\text{Absortion } (\%) = \frac{(\text{Difference in Weight})}{(\text{Original Weight})} \times 100\% \tag{2}$$

2.6 Mineralogical and Chemical Analysis

The mineralogy of marble waste and geopolymerized concrete were identified and quantified by the X-ray diffraction (XRD) analysis. The marble waste samples were prepared into fine particles (<4mm) for the XRD analysis. Samples were then tested with Bruker AXS Germany D8 Advanced X-ray diffractometer device at an angle of 1°/min(0.025^o) and a spectrum of 0.2s/s over a 5-50^o disperse distance. Meanwhile, scanning electron microscopy (SEM) machine (model Hitachi SU3500, Tokyo, Japan) was utilized to identify the surface morphological structure of the mineral components in the samples. The surface of the samples was observed in the secondary electron (SE) image. X-ray fluorescence analysis (XRF, Rigaku Primus IV) was performed to examine the chemical composition of the materials in which the results were obtained in the percentage of weight (wt.%).

3. Results and Discussion

3.1 Mineralogical and Chemical Properties of Marble Waste

Mineralogical composition is an important criterion to determine the suitability of mining waste as supplementary cementitious material [16], [17]. Three major minerals are found in marble waste composition. The mineral composition is determined through XRD analysis. Table 2 shows the three major minerals identified and their compositions in the marble waste sample. Calcite, $CaCO_3$ is the most abundant mineral found in marble waste composition. As calcite is the main mineral that forms marble, it should be the major mineral composed in marble. It is the major amount of mineral found exceeding 30% of the total mineral composition. Moreover, huge amounts of calcite in marble usually appear in white color. Akemanite, $Ca_2MgSi_2O_7$ and bustamite, $(Ca,Mn)SiO_3$ are the other two minerals. $Ca_2MgSi_2O_7$ and $(Ca,Mn)SiO_3$ are calcium-based minerals. Both minerals are identified in minor amounts which is between 2-10% of total marble waste composition. Thus, mineralogical properties of marble waste are dominated by calcite and calcium based minerals. Calcium carbonate powder can act as filler and enhance the rate of cement hydration and material strength produced at the initial stage [18]. The use of calcite in the concrete production comes with benefits. Calcite can significantly affect concrete production particularly on the permeability of the concrete produced and also the chemical structure [18].

Table 3 shows the chemical composition of marble waste and OPC. Both are done by XRF analysis. Calcium oxide (CaO) was found to be a major chemical in both marble waste (56.50%) and OPC (63.17%). Other than CaO, silicon dioxide (SiO) was also found in marble waste and OPC. CaO was found to be the major chemical composition of marble waste because marble waste is dominated by calcite and calcium based minerals. Marble waste can be approved as SCM as it is high in calcium and silicate where it is comparable with OPC. Aside from CaO, all other chemicals are found in smaller amounts.

Table 2 *Mineral composition of marble waste*

Minerals	Chemical Formula
Calcite+++	$CaCO_3$
Akemanite+	$Ca_2MgSi_2O_7$
Bustamite+	$(Ca,Mn)SiO_3$

+++ Major composition (>30%); ++ Moderate amount (10-30%);
+ Minor composition (2-10%) (Minerals analysis by XRD)

Table 3 *Chemical composition in wt. % of marble waste and ordinary Portland cement*

Chemical composition*	Marble waste	Ordinary Portland Cement
CaO	56.50	63.17
SiO_2	0.73	19.98
Al_2O_3	0.51	5.17
MgO	0.45	0.79
Fe_2O_3	0.53	3.27
ZnO	0.29	<0.01
BaO	0.05	<0.01
K_2O	0.04	<0.01
SrO	0.03	<0.01
P_2O_5	0.02	<0.01
SO_3	0.02	<0.01
LOI	40.8	2.4
Pozzolanic oxide$^+$	1.77	28.42

*Chemical composition as identified from XRF

LOI – Loss on ignition; $^+$Pozzolanic oxides (Sum of $SiO_2 + Al_2O_3 + Fe_2O_3$)

3.2 Compressive Strength of Geopolymerized Concrete

The compressive strength of geopolymerized concrete specimens were tested, which relies on their concrete design. The three main components of the concrete design were marble waste, AAM and OHE. Based on Fig. 2, the average compressive strength of B1 and B3 were higher than the other concrete specimens, particularly the control specimens. C1 was the control for normal concrete without any geopolymerization method (marble waste, AAM and OHE). Meanwhile, C2 only excluded AAM and C3 excluded OHE. However, the highest compressive strength in 28 days curing was shown by sample B1 with 6.04 N/mm^2. B1 contains marble waste as SCM, 0.4 AAM/cement ratio and 3 days OHE. Both C1 and B1 showed an increasing trend of compressive strength. The lowest compressive strength was 3.02 N/mm^2. The lowest compressive strength specimen was detected in the 14 days curing with B3 concrete design. B3 contains marble waste as SCM, 0.3 AAM/cement ratio and 5 days OHE. It is believed that a larger degree of reaction can be found in concrete with longer OHE [8]. As noted earlier, the composition of marble waste was limited to 20% in this study. It has been observed that at 20% of cement replacement, there is a slight fall of compressive strength in the concrete production [19].

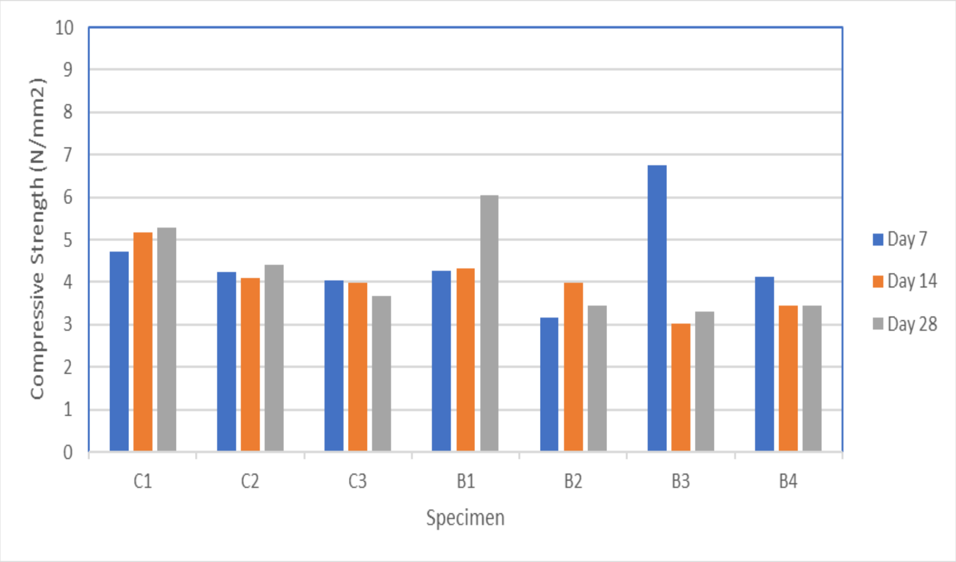

Fig. 2 *Compressive strength of concrete specimens*

Reduction in compressive strength can happen due to some reasons. It can be due to the increase in porosity and non-homogenous mix of the concrete specimen [20]. As the concrete type is masonry, the low compressive strength can be expected. Hence, normal concrete showed a good early development of strength. Meanwhile, the geopolymerized concrete can have good final compressive strength after 28 days of curing. The best concrete design was B1 (marble waste, 0.4 AAM/ cement ratio and 3 days heat period) and B3 (marble waste, 0.4 AAM/ cement ratio and 3 days heat period) because it showed an increasing trend throughout 28 days and achieved a higher average of compressive strength than C1 (control specimen). Therefore, OHE can provide a better result in terms of concrete compressive strength. Heating can be a good method for curing the geopolymerized concrete in terms of early development of strength. Geopolymerized concrete is usually hardened between 60°C to 90°C [21]. The use of OHE can elevate the temperature better causing the formation of larger pores within the specimen which can improve the strength of the concrete [6]. The pores are formed because the water vaporizes due to heat exposure. Furthermore, a higher Si/Al ratio of cement matrix may produce a strong concrete [8]. This is essential to develop compressive strength of geopolymerized concrete products.

3.3 Water Absorption of Geopolymerized Concrete

Strength of aggregate can be determined through water absorption. Higher water absorption means that concrete is more porous and has lower strength. Water absorption has an important role in the durability of concrete [15]. However, the lack of a clear correlation between internal and surface water absorption and compressive strength suggested that strength cannot be solely determined by water absorption [22]. Fig. 3 shows the highest differences in water absorption from day 7 to day 28 is from B3. It shows the highest water absorption recorded on day 7 (15.02%) and lowest water absorption recorded on day 28 (12.29%). B3 contains marble waste as SCM replacing one part of sand, 0.3 AAM/cement ratio and 5 days OHE. All of the specimens show a decreasing trend of water absorption throughout 28 days. As anticipated, water absorption was reduced with increasing curing time. Regardless of any specimens, the water absorption in 28 days was below the 25% limit.

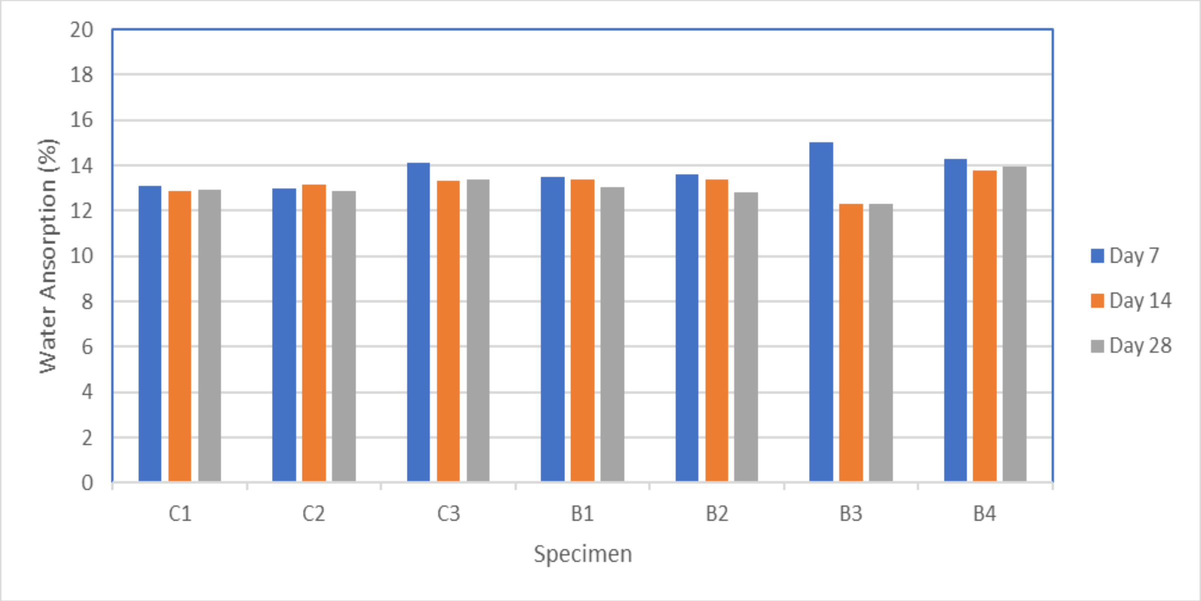

Fig. 3 *Water absorption of concrete specimens*

3.4 Effect of AAM on Compressive Strength

The use of marble waste can be validated with alkaline activation to improve the physical and mechanical properties of the concrete produced [23]. C2 is the control for the effect of AAM on compressive strength. C2 uses two components of the geopolymerization method (marble waste and AAM). On average, B1 and B3 have higher compressive strength than the control (C2) as shown in Fig. 4. Specimen B1 has the highest compressive strength with the mean of 4.88 N/mm^2. B1 consists of marble waste as SCM, 0.4 AAM/cement ratio and 3 days OHE. Meanwhile, specimen B3 has a mean compressive strength of 4.36 N/mm^2. B3 uses marble waste as SCM, 0.3 AAM/cement ratio and 5 days OHE. Exceptionally high strength was achieved at 28 days (for B1) and at 7 days (for B3). However, there were no statistically significant differences in the compressive strength between C2, B1, B2, B3 and B4. It might be due to the low strength cementitious material and masonry type of concrete-based material produced in this study. Thus, the use of marble waste with alkali activation material may result in good effects on physical and mechanical properties of concrete [24]. Marble waste which is known to contain aluminosilicate is suitable to be used as precursor with alkali activator. This is because the synthesis of reaction when using alkali activator in liquid form can create amorphous to semi-crystalline silicate and aluminate polymeric networks which can improve strength development [7].

3.5 Effect of OHE on Compressive Strength

It is known that heating can increase the compressive strength of geopolymer concrete [25]. C3 acts as a control for the effect of OHE on compressive strength. C3 uses two components of the geopolymerization method (marble waste and OHE). On average, B1 and B3 have higher compressive strength than the control (C3) as seen in Fig. 5. Specimen B1 has the highest compressive strength with the mean of 4.88 N/mm^2. B1 consists of marble waste as SCM, 0.4 AAM/cement ratio and 3 days OHE. Meanwhile, specimen B3 has a mean compressive strength of 4.36 N/mm^2. B3 uses marble waste as SCM, 0.3 AAM/cement ratio and 5 days OHE. Exceptionally high strength was achieved at 28 days (for B1) and at 7 days (for B3). There are no statistically significant differences in the compressive strength between C3, B1, B2, B3 and B4. It might be due to low-strength cementitious material and masonry type of concrete-based material. As noted earlier, the development of concrete strength is dependent on the state of curing [6]. OHE curing can help in strength development at an early age concrete, while facilitating the pore development which can improve the concrete strength.

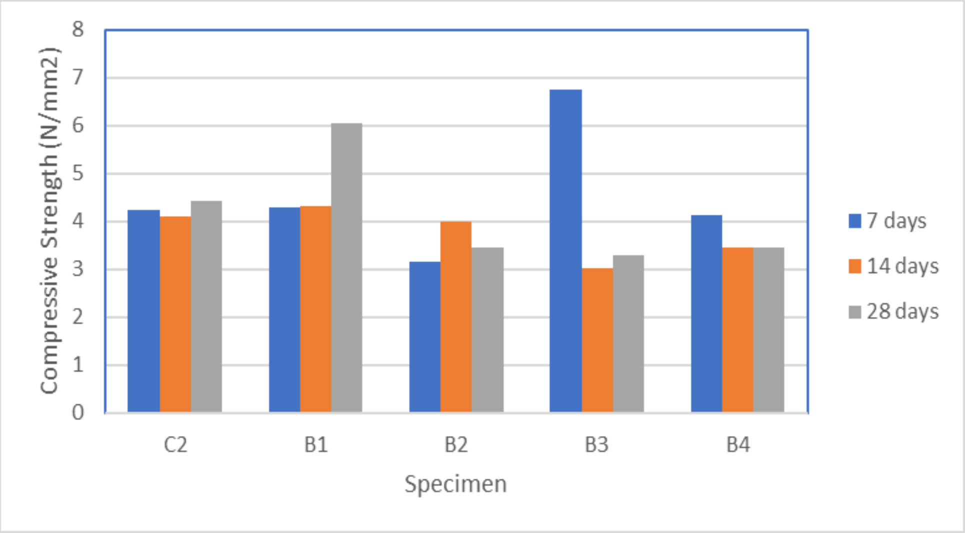

Fig. 4 *Effect of AAM on compressive strength*

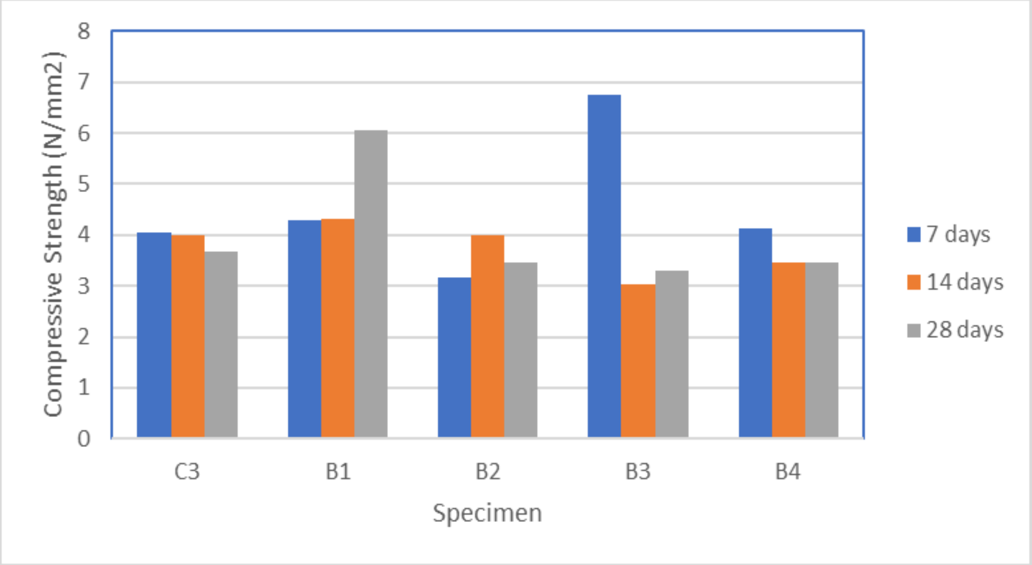

Fig. 5 *Effect of OHE on compressive strength*

3.6 Mineralogical Properties of Geopolymerized Concrete Product

The use of marble waste as SCM plays an important role in the mineralogical composition of the geopolymerized concrete. Mineralogical composition is important to identify the formation of geopolymerized minerals in the specimen [26]. Based on Fig. 6, the XRD diffractogram shows that the highest peak of calcium magnesium carbonate signified the major mineral that was found widespread in the geopolymerized concrete. The marble waste influenced the mineral composition of the geopolymerized concrete as the raw material contains large amount of calcium-based minerals. For instance, calcium magnesium carbonate was found in the geopolymerized concrete as much as 64.9% of the total composition. Besides, calcium disilicate also shows a high amount of composition (20.5%). Meanwhile, iron ore hydroxide (8.6%), aluminum silicon titanium (3.0%) and potassium tin oxide (2.9%) compose a minor proportion of the concrete. The production of calcium disilicate or calcium silicate hydrate in the specimen proves that geopolymerization was formed [27]. It was found that a greater composition of calcium disilicate (28.3%) was formed with 3 days OHE and 0.4 AAM/cement ratio. Meanwhile, the SEM image in Fig. 7 shows the rhombohedral shapes supporting the XRD diffractogram of geopolymerized concrete [28]. The small elongated shapes represent calcium silicate hydrate production. It shows the development of calcium silicate hydrate, C-S-H, the main cementitious product in the material.

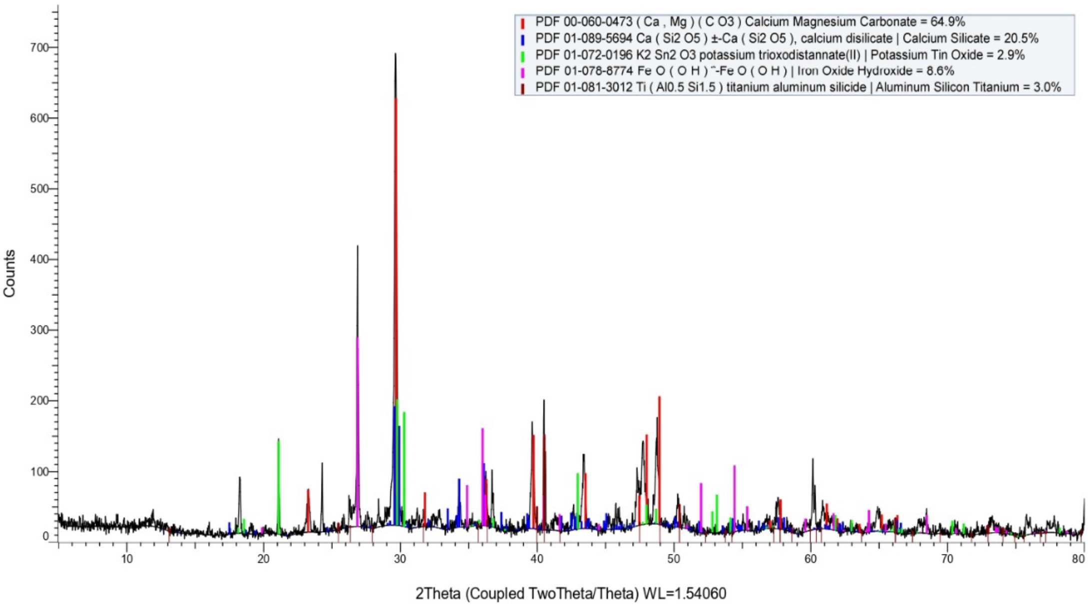

Fig. 6 *XRD diffractogram of geopolymerized concrete*

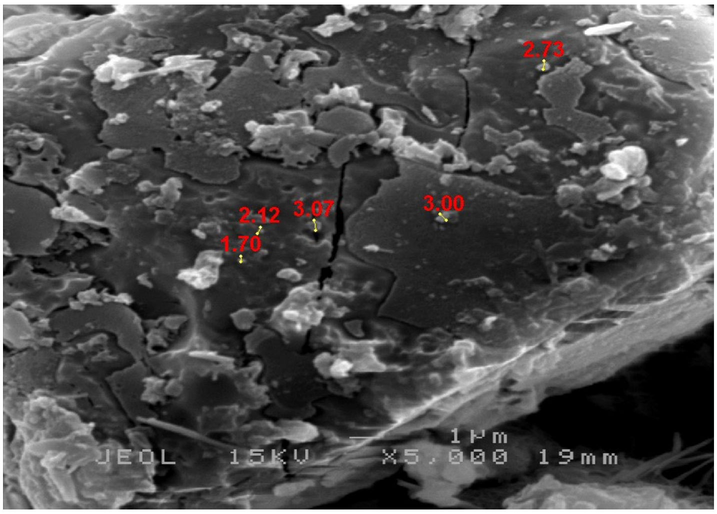

Fig. 7 *SEM images of geopolymerized concrete at magnification of 5.00 K*

4. Conclusions

Marble waste, alkali activated material (AAM) and outdoor heat exposure (OHE) were incorporated into the design of geopolymerized concrete and the effects of concrete designs on the properties of geopolymerized concrete were investigated. Calcite was found to be the major composition of minerals in marble waste samples with more than 30% of total composition. On average, B1 and B3 showed a higher compressive strength than the control specimens (C1, C2 and C3). The best concrete design was B1 (0.4 AAM/cement ratio and 3 days heat period) and B3 (0.3 AAM/cement ratio and 5 days heat period) as it showed an increasing trend throughout 28 days and achieved higher compressive strength than C1 (control as normal concrete). It was found that a greater composition of calcium disilicate was formed within 3 days OHE and 0.4 AAM/cement ratio. Thus, this study has shown that marble waste can be utilized as a supplementary cementitious material while incorporation of AAM and OHE in the concrete design will improve its strength performance. Findings would be useful to tackle the issues of waste production from mining industry while producing green construction material that is sustainable to the environment.

Acknowledgement

This work was supported by the research grants provided through the Fundamental Research Grant Scheme, FRGS/1/2023/TK08/UPM/02/9 (5540604), Ministry of Higher Education Malaysia (MOHE) and Universiti Putra Malaysia, IPS Grant 9709500.

Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Mohamad Adib Aiman Mohd Montare, Faradiella Mohd Kusin;* ***data collection:*** *Mohamad Adib Aiman Mohd Montare;* ***analysis and interpretation of results:*** *Mohamad Adib Aiman Mohd Montare, Muhammad Amir Aizat Khamis, Umi Rukiah Abdullah;* ***draft manuscript preparation:*** *Mohamad Adib Aiman Mohd Montare, Muhammad Amir Aizat Khamis, Faradiella Mohd Kusin. All authors reviewed the results and approved the final version of the manuscript.*

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