

EFFECTS OF METAKAOLIN ON COMPRESSIVE STRENGTH AND PERMEABILITY PROPERTIES OF PERVIOUS CEMENT CONCRETE

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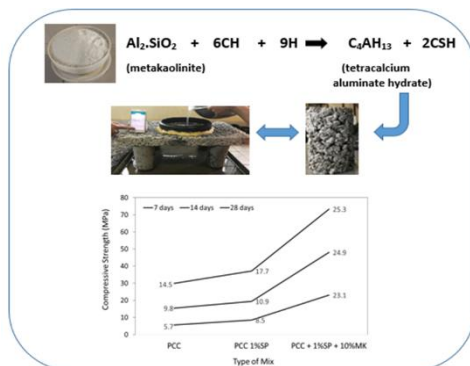
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Graphical abstract



Abstract

This paper presents the experimental results on the effect of metakaolin (MK) as a replacement of cement in pervious cement concrete based on compressive strength, void ratio and permeability test. Metakaolin was used to replace Portland Cement Composite (PCC) in pervious concrete by 10% (by wt.). The results show that pervious concrete sample containing 10% metakaolin with 1% superplasticizer exhibited 4 times higher compressive strength at 7 days when compared to normal cement concrete. In addition, the continuous voids of 10%MK concrete sample was found higher than PCC only, indicating the effectiveness of metakaolin in improving the interfacial bond of the binders due to micro-filler effect of metakaolin and the additional CSH gel formation. The continuous voids of pervious concrete with 10% MK is 17%, which is higher than the percentage of continuous voids in normal pervious concrete. This finding is also supported by the infiltration rate of concrete samples. Furthermore, the microstructure analysis through Backscattered Electron Imaging and X-Ray Diffraction reported denser matrix and CH reduction in polished paste samples due to 10%MK addition.

Keywords: Metakaolin, pervious concrete, compressive strength, voids, infiltration

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1.0 INTRODUCTION

The study on "green" sustainable materials by reusing by-product materials in construction has been increasing to solve environmental issues related to global warming. One of the studies is the inclusion of supplementary cementitious materials to reduce the amount of cement in mortar or concrete. Cement as the main material in concrete production has been minimized by replacing it with pozzolanic materials such as fly ash, slag, silica fume and metakaolin. The replacement aims to reduce carbon dioxide content during cement production since 1 ton of cement production releases 1 ton of carbon dioxide to the atmosphere leading to undesirable climate change.

By producing blended cement materials, the reduction can be expected to be at least 5%-20% of total CO₂ emissions [1].

The use of supplementary cementitious materials can be applied widely in making concrete elements including normal and high performance concrete. Based on some previous studies, the mechanical and durability properties of normal cement concrete has been improved due to the inclusion of supplementary cementitious materials. Recently, the application of pervious concrete has been discussed by scholars through some researches to improve its properties.

Pervious concrete is known as concrete with near zero-slump with little or no sand and consisting of cement, coarse aggregate, admixtures and water

(ACI 522R-10). These ingredients will form hardened concrete with typical connected pores of 2 to 8 mm that allow water to pass through. The void content of pervious concrete is about 15% to 25% with a flow rate of 0.34 cm/second [2]. Some factors that are generally effect the characteristics of pervious concrete are the optimum value of water/cement ratio, the use of admixture, aggregate sizes, and aggregate/cement ratio [3].

The application of pervious concrete is mainly used for parking lots, rigid drainage layers, structural wall application, pavement, surface courts, bridge embankments, green houses, pedestrian walkways etc. Other advantages of pervious concrete on environmental issues are to recharge groundwater and reduce stormwater runoff [4]. On the other things, pervious concrete is suitable for natural aggregates resources preservation and energy savings due to low compaction needed during installation into a pavement construction [5]. The use of pervious concrete also reduces tire-pavement interaction noise and enhances the skid resistance [6]. However, due to the pore structure size of pervious concrete is larger than typical concrete, therefore, low strength can be expected.

Concerning the minimum mechanical strength of pervious concrete, some experimental works have been conducted to utilize the use of pozzolanic materials in improving the properties of pervious concrete. Reference [2] studied the use of steel slag aggregate as by-products material from steel production and thermal power station to replace coarse and fine aggregates. In this study, cement was replaced by silica fume and fly ash. The results revealed the improvement of compressive and flexural strength due to the inclusion of waste materials. In another study, the use of fly ash to replace cement in pervious concrete as a pavement material have been evaluated as in [7] and [8]. The optimum mix proportion was found in mixes with 10 and 20% replacement of cement by fly ash with 10% and 15% fine aggregate. On their extension work, it was also found the reduction of total voids in pervious concrete was around 12-16%. From this study, the potential application of pervious concrete with fly ash addition can be for sub-base/base layer in flexible and a sub-base in a rigid pavement. The other approaches such as reduction of aggregate/binders ration, incorporation of pozzolanic materials, the addition of sand or polymer modification of matrix have also been evaluated to improve the strength of pervious concrete.

Although the studies on pervious concrete using waste materials have been increasing, the experimental works on the inclusion of metakaolin as one of natural resources in improving the properties of pervious concrete is very less. Metakaolin is anhydrous solid material with rich silica and aluminate obtained by calcining kaolin clay at a specific temperature depended on the type of predominant clay mineral in the sample. In cement composites, metakaolin reacts with calcium hydroxide during hydration

process to form additional products of calcium silicate and calcium aluminate hydrates that are responsible for the strength development. The improvement of strength on metakaolin cement composites was also attributed to the high surface area and the platy shape of metakaolin particles [9]. Based on some other studies, the addition of metakaolin (depending on metakaolin type and some other properties such as w/c ratio and replacement level) is beneficial in increasing compressive strength, splitting tensile strength and elastic modulus values of concrete [10]. The percentage replacement of cement by metakaolin was found at 10%, where beyond of that level the strength values were reported comparable with the strength of typical concrete mixture [11-13]. The significant breakthrough in metakaolin mixtures was also found in durability properties of cement mortar and concrete. Reference [14] reported the positive effect of 10% and 15% metakaolin in cement concrete in improving the resistance of concrete on the chloride diffusion and sulfate attack. Furthermore, the objective of this study is to investigate the effectiveness of using metakaolin in pervious concrete mixtures based on compressive strength results, void ratio and permeability properties. The microstructure analysis is also reported based on Backscattered Electron Imaging and X-Ray Diffraction tests.

2.0 METHODOLOGY

Portland Cement Composite (PCC), coarse aggregate with maximum size of 20 mm, metakaolin and water are the main materials used in pervious concrete mixtures. Metakaolin was obtained by calcinating kaolin sourced from Toraget Village, Minahasa Region in North Sulawesi Province, Indonesia. The chemical composition of PCC and metakaolin are listed in Table 1.

Table 1 Properties of PCC and Metakaolin

Chemical Analysis	PCC (%)	Metakaolin (%)
SiO ₂	20.92	43.88
Al ₂ O ₃	5.49	38.79
Fe ₂ O ₃	3.78	0.42
CaO	65.21	0.91
MgO	0.97	0.92
K ₂ O	-	0.73
Na ₂ O	-	0.23

The calcination period to form metakaolin was conducted at the Institute for Research and Standardization of Industry in Manado for 6 hours with constant temperature of 800°C. In the mixture proportions, the optimum content of metakaolin used as a replacement of cement was selected to be 10% by wt. This is based on the highest compressive strength obtained from previous study conducted as in [13].

Table 2 shows the mixture proportions of pervious concrete containing metakaolin. The aggregate/cement (a/c) ratio = 2, with cement content and water/cement ratio was kept constant at 400 kg/m³ and 0.3, respectively. The effect of using superplasticizer was also observed based on the compressive strength results to be compared with PCC sample without superplasticizer.

Table 2 Mixture proportions of pervious concrete containing metakaolin in Kg/M³

Type of mix	C ^a	M ^b	CA ^c	W ^d	SP ^e
PCC	400	-	800	120	4
PCC 1% SP	400	-	800	120	4
PCC+1% SP+10% MK	360	40	800	120	4

^aC=Cement; ^bM = Metakaolin; ^cCA=Coarse Aggregate; ^dW=Water; ^eSP=Superplasticizer

All concrete samples with 100 mm x 200 mm cylinder size were mixed in a pan mixer and demoulded after 24 hour. The samples were then cured in a water at room temperature until the testing days. In this experiment, three types of mixes are selected to investigate two parameters which are the effect of adding 1% superplasticizer and influence of metakaolin inclusion as a supplementary cementitious material. The pervious concrete samples before testing can be seen in Figure 1.



Figure 1 Pervious concrete samples

A. Compressive Strength

The compressive strength test of pervious concrete with and without 10% metakaolin was conducted based on ASTM C39 standard [15]. Concrete samples were tested after water curing at 7, 14, and 28 days. The compressive strength values were reported based on the average of three concrete samples tested using the Universal Testing Machine as seen in Figure 2.



Figure 2 Compressive strength test set-up

B. Void Ratio

This test was proposed by Japan Concrete Institute (JCI) and conducted in order to calculate the total void ratio that is defined as the percentage of the total volume of voids to the total volume of the specimen [16]. In this test, the concrete cylinder specimens with size of 100 x 200 mm were prepared after curing at 7 days. The volume of specimens was measured as V_1 . After water curing, the mass of samples in water was recorded (W_1). The samples were then left for 24 hour at room temperature and then weighted as W_2 . On the next day, the measurement in water was taken again (W_3) to calculate the total void ratio (A_t) based on the formula in Equation (1):

$$A_t (\%) = 1 - \frac{(W_2 - W_1) / \rho_w}{V_1} \times 100 \quad (1)$$

Additionally, the continuous void ratio (A_c) can be also calculated using the formula in Equation (2):

$$A_t (\%) = 1 - \frac{(W_2 - W_1) / \rho_w}{V_1} \times 100 \quad (2)$$

C. Infiltration Rate

The infiltration rate was conducted using infiltration ring according to ASTM C1701 standard [17]. Total water of around 18.0 ± 0.05 kg was poured into the ring and the elapsed time was recorded to the nearest of 0.1, started from the time when water impacted the pervious concrete surface until the free water disappeared from the surface (see Figure 3). The formula used to calculate the infiltration rate is shown in Equation (3):

$$I = \frac{KM}{(D^2t)} \quad (3)$$

Where:

- I = Infiltration rate (mm/h)
- M = Mass of infiltrated water (kg)
- D = Inside diameter of infiltration ring (mm)
- t = time required for measured amount of water to infiltrate the concrete (s)
- K = 4 583 666 000 in SI units



Figure 3 Infiltration rate test set-up

D. Scanning Electron Microscopy

This analysis was conducted using to evaluate the pores distribution of cement paste samples containing 10% metakaolin after 28 days of water curing based on Backscattered Electron Images. The cut paste samples were mounted in resin, polished using water free lubricant and covered by a layer of platinum to ensure that the samples were electrically conductive.

E. X-Ray Diffraction

This test was conducted to analyse the crystalline phases of cement paste samples based on its diffraction image. The sample for this analysis was prepared at approximately 3 grams of cement paste powder with particle sizes of 10-20 μm that grinded using laboratory porcelain mortar after curing days. The analysis was conducted at a speed of 0.5°/min with 2θ angle from 10° to 60° using D8 Advance Diffractometer (Bruker-AXS, for the cement paste sample prepared after 7 and 28 days of water curing. The voltage of and current used for the analysis were fixed at 40 kV and 30 mA, respectively. At every testing date, cement paste samples were cast and refined for the analysis.

3.0 RESULTS AND DISCUSSION

A. Compressive Strength

The compressive strength results of concretes at 7, 14 and 28 days can be seen in Figure 4 while Figure 5 shows the activity index of pervious concrete with variation of mixture proportions.

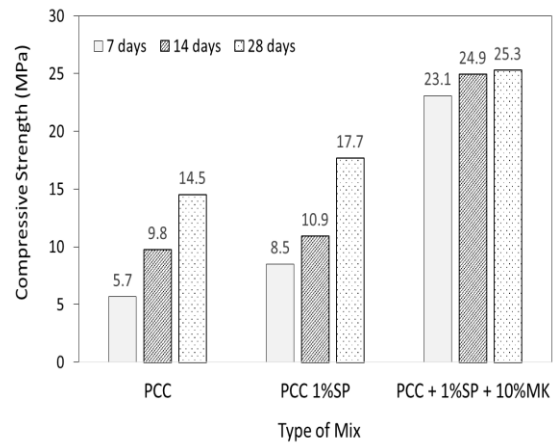


Figure 4 Compressive strength of pervious concrete at 7, 14, and 28 days

Based on the figure, it can be noticed that the compressive strength of pervious concrete is increasing as the curing age increased. When 1% superplasticizer was added into the mixture, the compressive strength of PCC concrete increased at about 32% higher at 7 days, 10% at 14 days, and 18% at 28 days, when compared to the compressive strength of PCC concrete without superplasticizer. The strength activity index shows that after adding 1% superplasticizer, the compressive strength of pervious concrete increases up to 12%, 22% and 50%, respectively, higher than typical PCC concrete at 7, 14 and 28 days (see Figure 5). These results show that the use of 1% superplasticizer as water reducer could balance the formation of pores and slightly improves the compressive strength of pervious concrete. However, increasing the dosage of superplasticizer could decrease the compressive strength since the flow characteristics might be also increased and loosen the binding characteristic between binders and aggregate.

In addition, the figure shows that there is a significant increase on compressive strength when 10% metakaolin was added to replace cement in pervious concrete containing 1% SP. The compressive strength of PCC+1%SP+10%MK at 7, 14 and 28 days is in the range of 23 to 25 MPa which is 2 – 4 times higher than the compressive strength of PCC concrete, as seen in Figure 5. This is an indication that the presence of 10% metakaolin significantly influences the formation of additional hydration product resulted in improving cement binder properties that is responsible for the improvement of compressive strength, particularly at the early age. However, the results of compressive strength do not show a clear trend with wide range difference since the values were taken from three concrete specimens only, therefore, it is important to cast more pervious concrete samples in order to find more exact results on the average of compressive strength.

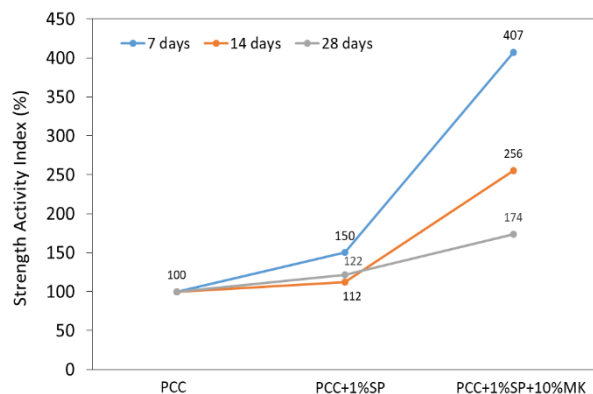


Figure 5 Strength activity index of pervious concrete at 7, 14, and 28 days

The high initial reactivity of metakaolin is also studied by reference [18] where the early age compressive strength development of cement paste with metakaolin was found better than the sample with silica fume. Moreover, the incorporation of metakaolin and the addition of superplasticizer is able to strengthen the interfacial transition zone (ITZ) between matrix and aggregate leading to densification and higher bond strength without neglecting the characteristic of pervious concrete in allowing water to penetrate through. The similar observation was also found on the study conducted by reference [13] when silica fume and superplasticizer were added into pervious concrete mixtures. Superplasticizer was found increased the fluidity of cement while silica fume at percentage of 5% by wt. improved the microstructure of pervious concrete.

B. Void Ratio

Concrete samples for void ratio were tested after water curing at 7 day. The percentage total voids, continuous voids and discontinuous voids of all type of mixes are presented in Table 3.

Table 3 Percentage of voids in PCC concretes containing 10% metakaolin

Type of mixes	Total voids (%)	Continuous voids (%)	Discontinuous voids (%)
PCC 1%SP	14%	12%	6%
PCC+1%SP+10%MK	19%	17%	2%

Based on the table, it can be seen that although the total voids of PCC containing 10% metakaolin is higher than the total voids of PCC, the PCC + 10%MK sample has more continuous voids which is about 17% with the discontinuous voids is 2%. This percentage indicates lesser discontinuous voids that was formed in PCC pervious concrete containing 10% metakaolin.

The similar observation was also reported by reference [19] where the presence of cementitious materials influences the increase of paste thickness that results in decreasing the porosity of the samples. Regarding to the effect of aggregate size on pervious concrete, reference [13] found that there was no significant change obtained on voids formation of pervious concrete prepared by different aggregate sizes. Therefore, it can be concluded that the presence of 10% metakaolin promotes denser microstructure due to its filler effect that makes less hardened voids on PCC pervious concrete and improves the strength properties.

C. Infiltration Rate

Table 4 presents the Infiltration Rate (I) of PCC Concretes Containing 10% Metakaolin. The water infiltration rate was conducted on the pervious concrete slab with size of 400 mm x 600 mm at 7 days. The rate of water infiltration represents the permeability value of pervious concrete with and without 10% metakaolin. Based on the experimental work, the infiltration rate of PCC concrete with and without 10% metakaolin is 12.73 mm/sec and 6.36 mm/sec, respectively, with the time required for water to infiltrate the concrete is 20 sec for sample without metakaolin and 40 sec with the inclusion of 10% metakaolin. The higher value of infiltration rate indicates the more porous of the concrete which affects the compressive strength of concrete. Based on the result, it can be explained that the addition of 10% metakaolin contributes the increase of cement paste thickness as well as the effect of cement paste containing metakaolin in filling the gap between coarse aggregate and improves the infiltration rate.

Table 4 Infiltration rate of PCC concretes containing 10% metakaolin

Type of mixes	M (kg)	D (mm)	t (sec)	I (mm/sec)
PCC+1%SP	18	300	20	12.73
PCC+1%SP+10%MK	18	300	40	6.36

D. Scanning Electron Microscopy (SEM)/ Backscattered Electron Image (BEI)

The microstructure images of polished PCC paste samples with and without 10% metakaolin taken from backscattered electron imaging at 28 days can be observed in Figures 6a-d.

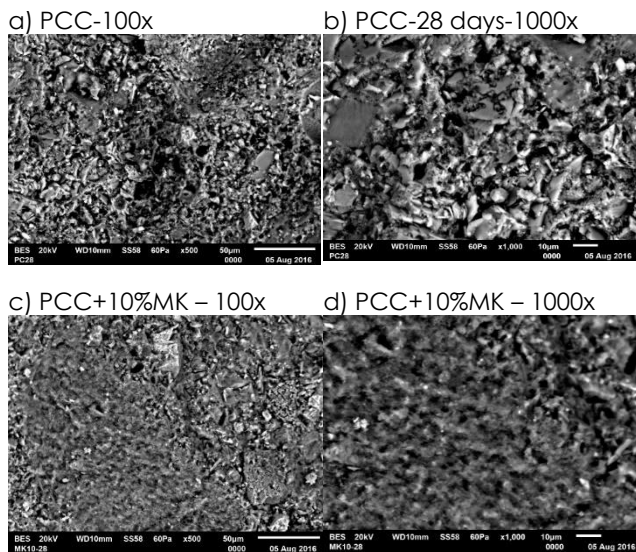


Figure 5 Backscattered electron images of a) cement paste at 50 μm , b) cement paste at 10 μm , c) cement paste with 10% MK at 50 μm , d) cement paste with 10% MK at 10 μm

In this study, two magnification levels of 100x and 1000x are considered to examine the pore segmentation based on the differential brightness in each images where the brightest represents the residual cement grains, followed by calcium hydroxide (CH), calcium silicate hydrate (CSH), and the black pores. It can be seen in the figures that PCC paste samples with 10% metakaolin has lesser black pores compared to PCC paste only. Additionally, more grey to dark areas can also be found indicating the effect of metakaolin in the formation of more CSH and denser microstructure when compared to the polished section of PCC paste. In another study, it was also reported that the consumption of calcium hydroxide or portlandite and the formation of additional CSH gel due to pozzolanic reaction of supplementary cementitious material refines the pore system in the matrix leading to a denser microstructure [20]. These results are then confirm the findings on compressive strength development due to 10% metakaolin addition, as explained earlier.

E. X-Ray Diffraction (XRD)

Figures 7a, b show the XRD patterns of hydrated PCC containing 10% metakaolin at 7 and 28 days. The peak intensities of calcium hydroxide is taken as a main indicator to evaluate the formation of CSH gel that responsible for strength development.

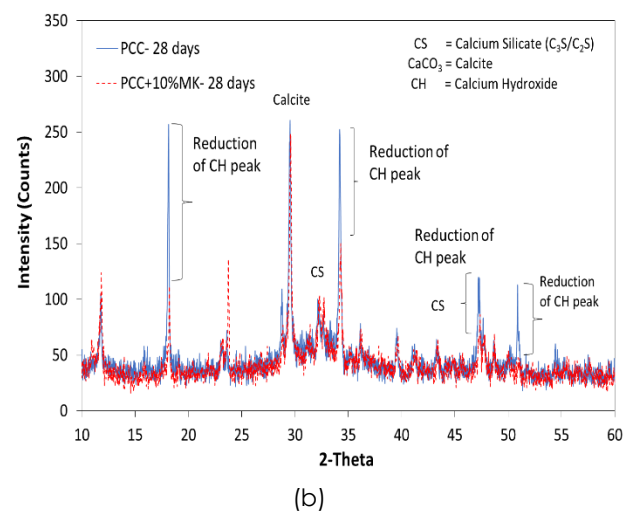
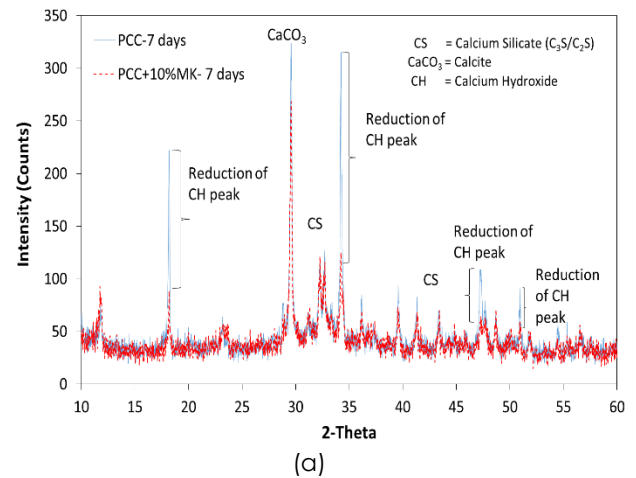


Figure 7 XRD peaks of PCC paste sample with 10% MK at a) 7 days and b) 28 days

It can be clearly seen in Figures 7a and b that the peaks of CH phase in paste containing 10% MK are lower than the peaks in control PCC paste after water curing at 7 and 28 days. The reduction of CH peaks after 10% MK addition can be seen at 18°, 34°, 47° and 51° of 2-theta scale with significant reduction of CH reduction can be seen at 18° and 34°. Moreover, at 29° of 2-theta scale, the peak intensity of calcite as a compound originated from cement and MK hydration is found lower in PCC+10%MK sample compared to control PCC sample due to lesser cement content after replaced with metakaolin. It is seen that the peak intensity of calcite decreases as the increase of curing time up to 28 days (see Figure 7b) while the peak intensities of CS are still present. Comparing the XRD peaks on PCC paste with 10% MK, it is clear that the inclusion of MK particles with silicate minerals act as nucleation agent which significantly consume the formation of CH and therefore enhance the formation of CSH gel that increase the compressive strength pervious concrete. However, since the location of calcite is overlapped with that of CSH, the presence of CSH could not be exactly evaluated. Furthermore,

the XRD results in this experiment are in line with the experimental results studied by reference [21]. In this study, metakaolin was found accelerated the rate of hydration through its microfiller and nucleating agent effects, hence, improves the mechanical strength properties of pervious concrete.

4.0 CONCLUSION

The addition of 10% metakaolin with 1% superplasticizer increases the compressive strength of pervious concrete with 1% superplasticizer at aggregate/binders = 2 and w/c = 0.3. The significant improvement can be observed at 7 days where the compressive strength of pervious concrete with 10% metakaolin exhibited 4 times higher compared to the sample without metakaolin inclusion. In terms of the percentage of continuous voids formed in PCC pervious concrete, the presence of 10% metakaolin increases the continuous voids up to 17% compared to the sample without metakaolin. Additionally, the infiltration rate of PCC concrete with and without 10% metakaolin is 12.73 mm/sec and 6.36 mm/sec, respectively. The lower infiltration rate of pervious concrete containing metakaolin indicates lesser pores formation due to the filling and nucleating agent effects of metakaolin that increase the binding properties between cement paste and coarse aggregate. Based on the microstructure analysis, the addition of 10% metakaolin in PCC pervious concrete improved the density of polished surface cement paste matrix. The consumption of CH phase due to metakaolin addition is also confirmed by the results from XRD analysis. The inclusion of MK particles with silicate minerals act as nucleation agent which significantly consume the formation of CH and therefore enhance the formation of CSH gel that increase the compressive strength of pervious concrete.

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