



Compressive Strength, Temperature Performance and Shrinkage of Concrete Containing Metakaolin

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Abstract: The influence of metakaolin (MK) on concrete was investigated as a partial replacement of cement, which is liable for high content of CO₂ emission during the manufacturing process. The study aimed to assess the compressive strength, temperature performance and drying shrinkage of MK blended concrete. Different ratios of MK (5%, 10%, and 15%) as cement replacement, and two ratios of superplasticizer (SP), 1.5%, and 2% as cement weight were utilized in this study. The results indicated that using MK increased the compressive strength at 28th day. The improvement of strength is ascribed to the pozzolanic reaction and the packing effect of fine MK particles. MK blended concrete experienced a decrease in the temperature rise of heat of hydration (HOH) and low drying shrinkage when compared to the control concrete. The SP ratio does not have significant effect on the temperature of the concrete mixtures: however, using 1.5% SP slightly reduces the drying shrinkage of MK blended concrete compared to 2% SP. In conclusion, Incorporation of MK as partial cement substitute enhances the concrete strength, thus various types of concrete can be produced. Low temperature and drying shrinkage of MK blended concrete help in reducing thermal crack after casting process.

Keywords: Metakaolin, concrete, compressive strength, temperature, shrinkage

1. Introduction

Concrete, the most widely used construction material in the present industry, consumes large quantities of cement and aggregates. The large amount of concrete is being consumed due to urbanization and its increasing demand globally, and that results in the development of high performance concrete and high strength concrete [1]. Cement is the most frequently utilized building ingredient in construction engineering. Cement is liable for high content of carbon dioxide (CO₂) discharge during the calcination process of cement production, and decomposition of calcium carbonate (CaCO₃) [2], [3]. The other pollutants emitted from cement industries like dust, nitrogen oxide and sulfur oxide are very dangerous to our environment [4]. These pollutant gases cause air pollution and lead to the greenhouse effect. In order to mitigate the negative effects on the environment, systematic solutions for safe and controlled concrete production need to be developed. For instance, using mineral admixtures such as MK could reduce the cement in concrete as one of the largest CO₂ sources and energy-consuming manufacturing.

Previous studies reported that the strength performance of concrete is negatively affected by employing 100% ordinary Portland cement (OPC) due to shrinkage and HOH issues [5]. Large amounts of OPC in concrete results in high heat evolution during the hydration process and temperature differential between the concrete core and surface,

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thus thermal cracking takes place [6]. Therefore, an effective solution should be developed to offset the heat emitted during cement hydration. The drawbacks of using 100% OPC in concrete can be overcome by replacing OPC by pozzolanic materials that enhance the performance of concrete [7]. MK is an artificial pozzolanic admixture obtained from calcination of clay mineral called kaolin [8]. The Malaysia's states have a huge quantity of kaolin material produced from active kaolin mines [9]. As a result, due to the abundance of kaolin resources, Malaysia can take advantage of the possibility to make eco-concrete by employing MK as a pozzolanic material in concrete manufacturing. MK is produced at a lower temperature compared to cement without emitting CO₂ [10]. MK is manufactured by heating kaolin in order to release the hydroxyl ions at temperature ranging from 650°C to 700°C [11], [12]. Increasing replacement levels of MK in concrete requires increased amount of SP due to high specific surface of MK [13]. In addition, MK is characterized by a high pozzolanic reaction and its capacity to accelerate cement hydration which aids in improving early strength [14], [15]. Particularly, MK containing a considerable amount of amorphous alumina/silica was proved to be potential in high strength concrete due to pozzolanic activity [11], [16]. Due to high aluminate content, MK is capable in the capacity of concrete to retard chloride penetration [17].

This study aims at testing the effect of replacing OPC with different ratios of MK in concrete and two different ratios of SP. The compressive strength, temperature of HOH and drying shrinkage of concrete were evaluated in accordance to each level of MK and respective SP.

2. Materials Utilized in the Concrete Mixtures

2.1 Metakaolin

MK is an aluminosilicate pozzolanic material obtained from calcination of clay mineral called kaolin in the form of white powder [18]. The calcination process modifies the particle structure making MK amorphous and highly reactive [4]. MK is manufactured by drying kaolinite clays at 105°C for 48 hr and thereafter, grinded in a ball mill for 40 min. The powdered clays then calcined in a furnace at 700°C and rate 10°C/min for 1 hr [19].

MK was used as supplementary binder with an average particle size of approximately 2.1 μm, as seen in Fig. 1(a). The particle size distribution and the specific surface area of MK are shown in Table 1.

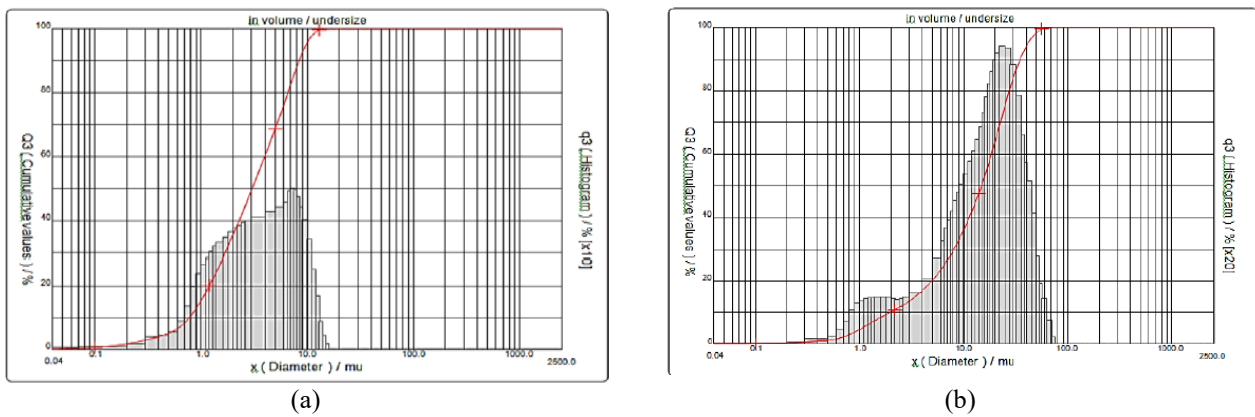


Fig. 1 - Particle size analysis for (a) MK, and; (b) OPC

Table 1 - Particle size distribution and specific surface area of MK and OPC

	Diameter at 10% μm	Diameter at 50% μm	Diameter at 90% μm	Specific Surface Area (cm ² /g)
MK	0.78	2.1	8.4	16156
OPC	1.94	11.52	34.8	5267

2.2 Cement

OPC manufactured in Malaysia has been used in this study. The cement conforms to the specification of BS 197-1:2000. OPC has an average particle size of approximately 11.52 μm, as seen in Fig. 1(b). The particle size distribution and the specific surface area of OPC are shown in Table 1.

2.3 Aggregate

Natural river sand was used as fine aggregate with particle size that passed sieve 5 mm. The used coarse aggregate was gravel, and the nominal maximum aggregate size is 10 mm. Sieve analysis was carried out to obtain the grading of fine aggregate and coarse aggregate that were later utilized in the concrete mix as shown in Fig. 2.

2.4 Water

Water that is clean and free from impurities from the water supply system was used to prepare the concrete mixes. Reaction of water with cement enables the chemical reaction contributing to the strength of concrete.

2.5 Superplasticizer Admixture

SP admixture of high efficiency in reducing mixing water was used to maintain the workability of fresh concrete. In this study, Dynamon NRG 1030 white colored liquid was utilized.

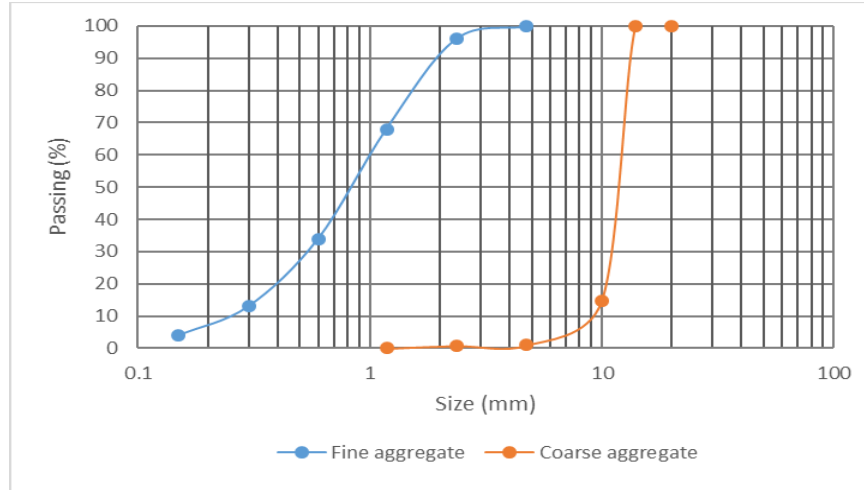


Fig. 2 - Sieve analysis of fine aggregate and coarse aggregate

3. Concrete Mixes

The proportions of the concrete mixes are shown in Table 2. The concrete mix proportions used throughout this study were designed based on the recommendation of the previous study [20]. For the control mix, the ratio for cement, fine aggregate and coarse aggregate was (1: 1.35: 1.88) respectively, and W/C ratio was kept constant at 0.27 for all mixtures. Eight mixtures were utilized incorporating MK (5%, 10%, and 15%) as cement replacement, and two dosages of SP (1.5% and 2%) as cement weight were added for each level of MK.

The concrete components were mixed for at least 10 minutes in a drum mixer to reach homogeneous state ensuring materials are correctly proportioned with no possibility to be segregated. Upon the completion of mixing, cubes with size of (100×100×100) mm for compressive strength test and prisms with size of (100×100×500) mm for temperature and shrinkage tests size were prepared. Then, the samples were demoulded after 24 hrs and cured in water at ambient temperature and humidity until the testing time.

Table 2 - Mix design proportion (kg/m³)

Mix	OPC	MK	FA	CA	W/b	SP (%)
REFSP1.5	550	0	742.5	1034	0.27	1.5
REFSP2	550	0	742.5	1034	0.27	2
MK5SP1.5	522.5	27.5	742.5	1034	0.27	1.5
MK5SP2	522.5	27.5	742.5	1034	0.27	2
MK10SP1.5	495	55	742.5	1034	0.27	1.5
MK10SP2	495	55	742.5	1034	0.27	2
MK15SP1.5	467.5	82.5	742.5	1034	0.27	1.5
MK15SP2	467.5	82.5	742.5	1034	0.27	2

OPC: ordinary Portland cement, MK: metakaolin, FA: fine aggregate, CA: coarse aggregate, SP: superplasticizer

4. Testing Methods

4.1 Compressive Strength Test

A compressive strength test was carried out to measure the maximum stress of hardened concrete. Three samples of (100 × 100 × 100) mm cube from each mixture were tested in the 28th-day and the average compressive strength

was recorded. The test was carried out according to BS 12390-3, using compression test machine with capacity of 3000 kN and loading rate of 7.0 kN/s. The maximum stress is calculated as seen below:

$$\sigma = \frac{P}{A} \quad (1)$$

where σ = normal stress, P = normal crushing force, and A = surface area.

4.2 Temperature Performance Test

The thermal characteristics of concrete are critical in the pre-hardening stage. The temperature rise is generated due to heat release within the concrete mixture during the hydration of cement. Studying the temperature performance is of great concern for mass concrete, notably during casting stage. The hydration process in concrete is characterized by the heat released during the exothermic chemical reaction [21]. The behavior of the HOH is different for each concrete based on the chemical composition of cementitious materials [22]. $\text{Ca}(\text{OH})_2$ can represent the hydration extents during the chemical reaction between water and cement [23]. The factors influencing heat development include cement fineness and weight, water/cement ratio, ambient temperature, mineral and chemical admixtures, and volume of concrete member [24]. Furthermore, the temperature of concrete is largely controlled by materials, mix properties and environmental factors [25], thus the heat may be lost or gained from the surrounding environment.

The temperature of the HOH test was carried out using a thermocouple (type K). Immediately after pouring concrete into the prism, the temperature sensor was inserted 50 mm deep into center of prism surface. The temperature values of concrete were monitored every 1-hour for 5days (120 hrs).

4.3 Drying Shrinkage Test

One of the main challenges of concrete pouring is preventing cracks during hardening due to volume change. Shrinkage occurs as a consequence of withdrawal of water from the capillary pores by the hydration of cement [26]. Water/cement ratio has a great effect on shrinkage of cement concrete [27]. Shrinkage takes place in concrete in different forms: plastic, autogenous, and drying shrinkage. Shrinkage of concrete occurs once the hydration process starts between cement and water resulting in the reduction of mortar volume [28].

In particular, drying shrinkage was tested, by exposing the hardened concrete at ambient temperature and humidity. Drying shrinkage was determined for the concrete samples of (100×100×500) mm prepared according to BS ISO 1920-8:2009 by specifying the length change. Two studs were placed at the middle section 200 mm apart immediately after casting. The length change with respect to the original length was accurately measured daily for 7 days after 24 hrs from casting and at 28-day. The drying shrinkage for the concrete prism is calculated using Eq. (2).

$$\text{Drying shrinkage (\%)} = \frac{\text{Change of length} - \text{Original length}}{\text{Original length}} \quad (2)$$

5. Results and Discussion

5.1 Workability of Concrete

Table 3 shows the slump results of the reference concrete and MK blended concrete. There is a little improvement in the workability for 5% MK mix. The increase of MK has slightly lowered the slump in comparison to the control concrete. This is ascribed to the fineness of MK in concrete, which absorbs more water during concrete mix [29]. However, 2% SP enhanced the workability of MK blended concrete compared to 1.5% SP. SP helps in separating and dispersing the agglomerated binder particles by creating electrostatic repulsion. Therefore, SP enhances the flowability and viscosity of concrete via the large specific surface of MK (16156 cm^2/g) compared to OPC (5267 cm^2/g), which provides an additional lubricating effect for the fresh concrete [30].

Table 3 - Slump test results

Mix	Slump (mm)	
	1.5% SP	2% SP
Control sample	173	181
5% MK	175	184
10% MK	171	183
15% MK	169	177

5.2 Compressive Strength

The 28-day compressive strength concrete was improved with increment of MK; however, using 1.5% SP attained higher compressive strength than 2% SP. As observed in Fig. 3. MK15SP1.5 concrete recorded the highest compressive strength with the value of 78.5 MPa, while the compressive strength of MK15SP2 is 69.5 MPa. The improvement of strength was justified by the pozzolanic reaction of MK with calcium hydroxide resulting in calcium silicate hydrate (C-S-H). High alumina in MK helped in the formation of calcium aluminate silicate hydrate (C-A-S-H) during the early age of curing that contributes to concrete strength. Furthermore, the fine particles of MK enhanced the packing density of cement matrix. This is in line with the studies of [31] and [32] who indicated enhancement of compressive strength with increasing MK up to 20% as OPC substitute.

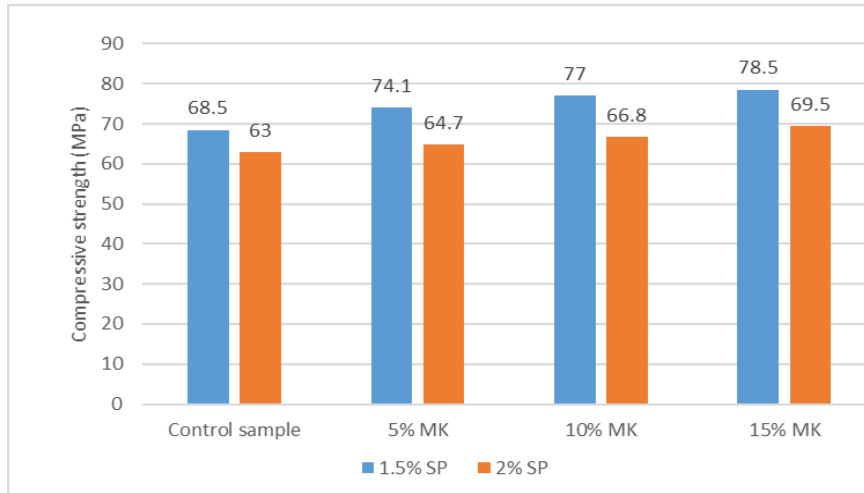


Fig. 3 - Compressive strength test results at 28-day

5.3 Temperature Performance

The temperature values of the HOH of concrete mixtures are shown in Fig. 4(a) and Fig. 4(b). The inclusion of MK with respective SP has changed the rate of temperature emitted from concrete. It is observed that the peak temperature of concrete containing 5-15% MK is lower than the control concrete, as shown in the graphs.

The analysis of the temperature data of concrete mixtures displayed a variation and fluctuation in temperature with time and led the concrete to reach the maximum temperatures at specific periods. The values for peak temperature, peak time and initial temperature are seen in Table 4.

Table 4 - Temperature performance of concrete mixes test results

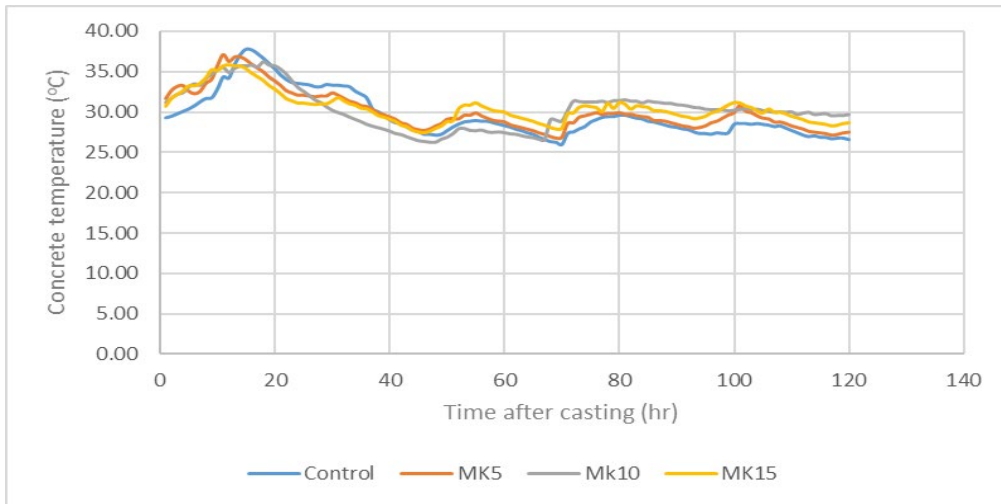
Mixture	Peak Temperature (°C)	Peak Time (hr)	Initial Temperature (°C)
RFSP1.5	37.8	15	29.30
RFSP2	39.1	20	30.30
MK5SP1.5	36.85	14	31.70
MK5SP2	38.20	15	29.40
MK10SP1.5	36	18	29.80
MK10SP2	37.30	15	31.50
MK15SP1.5	35.70	14	30.70
MK15SP2	36.50	18	29.60

High cement content of concrete contribute to the high hydration temperature [33]. M15SP1.5 and MK15SP2 concretes recorded the lowest temperature peak with values of 35.70 °C at 14 hrs and 36.50 °C at 18 hrs after casting respectively. This was confirmed by [34] stating that MK experienced a decrease in the temperature rise of HOH when compared to the control concrete. In addition, the temperature of the HOH may be accelerated by an ambient temperature.

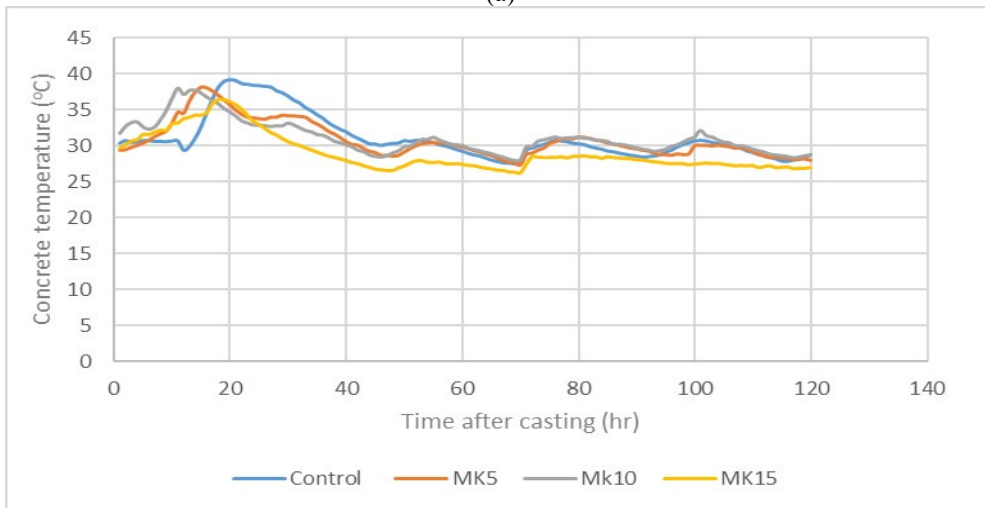
5.4 Drying Shrinkage

Fig. 5(a) and Fig. 5(b) show the behavior of drying shrinkage of prism specimens with incorporation of MK in concrete over 28 days. All the prism specimens exhibited drying shrinkage rapidly at an early stage. The drying shrinkage decreased with the increase of MK ratio in concrete in comparison to the control concrete. MK15SP1.5 and

MK15SP2 have achieved the lowest drying shrinkage. This is ascribed to the increase of concrete density and high particle packing with the incorporation of MK. The high fineness and the pozzolanic reactivity of MK might influence shrinkage magnitude [35]. This is in line with [36] study that MK can reduce the shrinkage at a lower W/C ratio. In addition, [37] observed low draying shrinkage of MK concrete compared to control concrete and mentioned that SP demand in concrete contributes to an increase in the drying shrinkage strains.

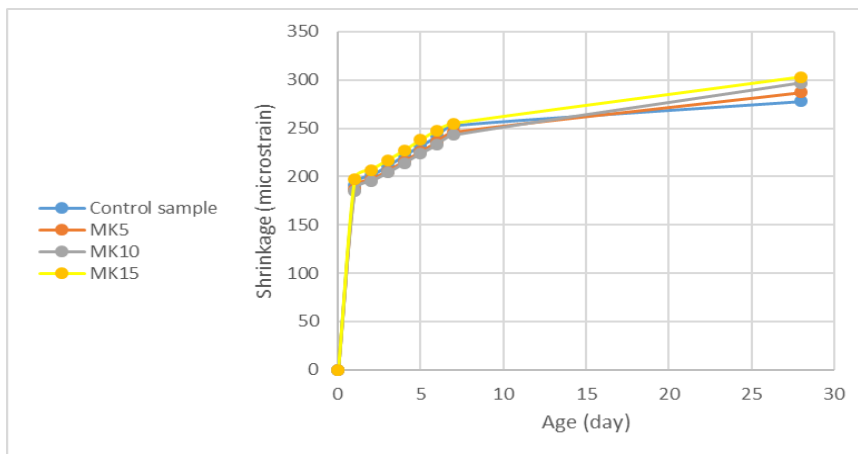


(a)



(b)

Fig. 5 - Temperature of concrete heat with varying ratios of (a) MK and 1.5% SP, and; (b) MK and 2% SP



(a)

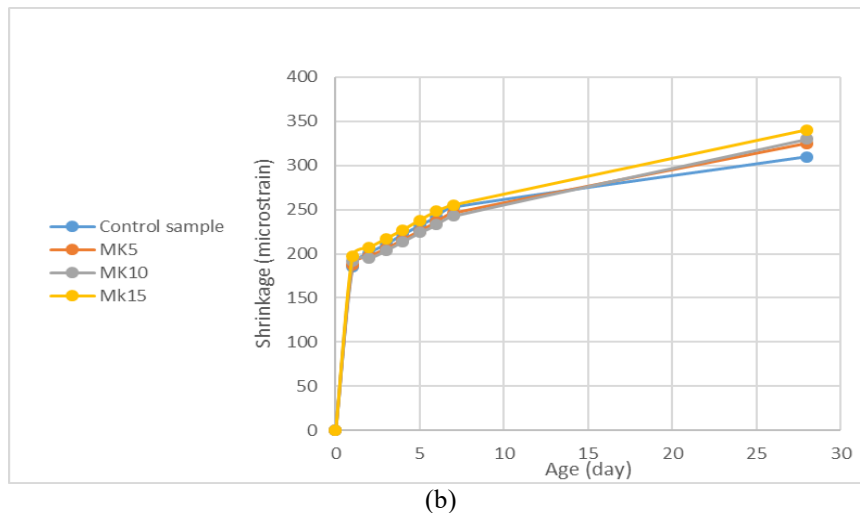


Fig. 5 - Shrinkage of mixes with different replacements of (a) MK and 1.5% SP, and; (b) MK and 2%SP

6. Conclusion

Based on all the tests results analysis, the effect of incorporating MK in concrete as partial replacement of cement on the compressive strength, temperature performance and shrinkage can be concluded as follows:

- Incorporating MK in concrete exhibited a considerable rise of compressive strength at 28 days particularly when using 1.5% SP. Consequently, MK could be incorporated in high strength concrete as blended cement.
- The peak temperature of concrete containing MK is less than control concrete. SP ratio has no significant effect on the concrete temperature. This indicates MK has an effective role in reducing the temperature of concrete during the hydration process. Eventually temperature reduction contributes to reduction of tensile stresses that is responsible for thermal cracking in concrete.
- Incorporating MK in concrete contributes to low drying shrinkage compared to the control concrete. 1.5% SP slightly reduces the drying shrinkage of MK blended concrete compared to 2% SP.
- MK can be utilized to promote production of sustainable concrete, which in turn minimizes the environment pollution.

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