

Influence of Steam Curing on the Performance of High-strength Concrete Incorporating Metakaolin

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Abstract

This study aims to investigate the influence of steam curing on the performance of high-strength concrete incorporating metakaolin. An experimental study was conducted to investigate the compressive strength, chloride permeability, initial surface absorption, and water absorption, as well as thermal degradation under various steam curing scenarios. Four concrete mixtures were prepared in this study, each with different percentages of metakaolin, as partial substitution of cement weight of 0%, 5%, 10%, and 15%. The water-binder ratio was set to 0.32 in all mixtures. The specimens underwent steam curing for 0, 4, 8, and 16 hours at a temperature of 55 °C. Results indicate that the specimens that have been steam cured and contained metakaolin exhibit improved compressive strength during the initial stages of their strength development. Steam curing has been demonstrated to enhance the concrete's resistance to chloride penetration and reduce initial surface absorption while increasing water absorption. The optimal replacement of cement with metakaolin to enhance strength and transport properties in high-strength concrete under steam curing regimes is between 10% and 15%. Future research could examine the effects of long-term steam curing on the durability and performance of concrete incorporating an optimal proportion of metakaolin.

Keywords

metakaolin, high-strength concrete, steam curing, industrial waste chloride resistance, differential thermal analysis

1 Introduction

Demands for construction materials, particularly cement, have remarkably increased due to the growing population and infrastructure development in many countries worldwide [1]. Cement manufacturing encompasses multiple stages, from stone crushing to cement bag filling [2]. During cement production, a substantial amount of energy is generated as heat, which is necessary for clinker processing. This procedure also releases a large amount of carbon dioxide (CO₂) into the atmosphere, as well as other greenhouse gases (GHGs). The urgency to mitigate the issues associated with cement manufacturing has been highlighted by increasing environmental and health concerns [3]. Concrete manufacturing and use, including its components and impacts on the environment throughout its lifespan must be analyzed from an environmental perspective [4]. The concrete industry experiences a decrease in cement use as part of the efforts toward sustainable growth [5]. Incorporating industrial and agricultural

waste materials into construction applications as supplementary cementitious material not only decreases the amount of cement used but also decreases risks related to health and the environment [6–8]. High-strength concrete is often characterized as concrete with a compressive strength of 60 MPa or more [9]. The production of high-strength concrete differs from traditional concrete preparation through the use of supplementary materials that include mineral and chemical compounds [10, 11]. Simultaneously, the manufacturing cost of producing high-strength concrete is greater than that of traditional concrete [12]. To minimize the expense of high-strength concrete manufacture while maintaining the concrete strength and durability, additional materials, specifically mineral admixtures such as silica fume, are used [13]. The global production of metakaolin, which is a highly effective supplementary cementitious material with pozzolanic properties, is substantial. It is commonly used to

decrease the amount of cement in concrete [14]. During the manufacturing of metakaolin, the quantity of CO_2 emitted is less than that during cement manufacture. In addition, limestone decarbonization in metakaolin production uses less energy. Metakaolin is regarded as an excellent cement substitute in the creation of cement-based matrices given its positive impact on the environment and its ability to address human health concerns [14, 15]. In addition to using metakaolin as a partial substitute for cement to produce cementitious concrete [16], it has been an essential component in the geopolymer (zero cement) concrete production [17]. Nevertheless, the use of significant amounts of industrial waste as a partial substitute for cement may result in a prolonged setting and hardening process, as well as a slower rate of concrete strength growth. Therefore, a decrease in compressive strength over the early ages (i.e., one to seven days) was evident when compared with the normal mixture [18]. To address this issue, a number of researchers have suggested implementing certain methods of steam curing or submerging in hot water for a significant duration and at appropriately elevated temperatures. This approach aims to improve the initial growth of compressive strength [19]. Steam curing accelerates the maturing process of cement-based products, thereby influencing their microstructural characteristics. This process impacts the development of concrete characteristics during the early stages [20]. Nevertheless, it could potentially exhibit adverse consequences at subsequent or later ages [21]. Several studies have reported the negative influence of steam curing on the mechanical and physical characteristics of concrete [22]. However, several studies have demonstrated that using pozzolanic materials as a partial substitute for Portland cement in concrete can effectively diminish the SO_3 level in the cement, mitigating the detrimental effects of delayed ettringite production [23]. However, increasing the early compressive strength of concrete through steam curing may influence other concrete properties, such as its chloride ion penetration resistance or chloride permeability [24]. To ensure concrete durability, its resistance to chloride ion penetration must be strengthened protect the concrete. The penetration of chloride ions into concrete initiates several chemical reactions between the chloride ions and the steel reinforcement. Metakaolin is usually a thermally activated aluminosilicate substance. The process involves subjecting kaolinitic clay to temperatures ranging from $650\text{ }^\circ\text{C}$ to $800\text{ }^\circ\text{C}$ [25]. Metakaolin, different from other manufactured pozzolans, such as fly ash and silica fume,

can be produced to satisfy high-quality standards. This approach ensures a consistent composition, higher purity, and improved reactivity when metakaolin is incorporated into concrete and reacts with calcium hydroxide (CH), a byproduct of the cement hydration process. This process alters the concrete's composition by increasing the amount of calcium silicate hydrate (C-S-H) gel [26]. The inclusion of metakaolin in concrete has demonstrated a substantial pozzolanic impact, resulting in significant enhancement in strength, particularly during the early stages of development. Metakaolin addition to concrete substantially improves the concrete's characteristics. Metakaolin nanoparticles effectively fill the spaces between the cement matrix, resulting in a more compact concrete. The probability of water and oxygen permeation is reduced by densifying the material, and the penetration of chloride ions, sulfate, and acid attacks is prevented. This method ultimately enhances the cement matrix's durability. Prior research has demonstrated that the addition of metakaolin resulted in a notable enhancement in the mechanical and durability, as well as the microstructural properties, of the concrete mixtures [5]. In the concrete construction industry, precast concrete plants are encouraged to achieve high early strength due to various factors, such as limited formwork, facilities, storage space, and time constraints. This condition allows faster removal of formwork and shorter curing period. The common goal is to expedite the hardening process of concrete by employing several techniques to achieve the appropriate strength level rapidly. Steam curing is a commonly employed approach for this objective. However, a review of previous literature reveals a paucity of studies on the effect of steam curing at temperatures of approximately $55\text{ }^\circ\text{C}$ and for periods of less than 24 hours on the properties of high-strength concrete containing metakaolin.

This study is significant because it establishes a precedent for investigating the impact of various steam curing scenarios on concrete that contains metakaolin at varying levels. The study investigated the effect of steam curing, applied over various period intervals, on the mechanical properties, transport characteristics, and microstructures of high-strength concrete. Therefore, an experimental study was conducted to examine the compressive strength, chloride permeability, initial surface absorption, and water absorption, as well as thermal decomposition analysis under different steam curing scenarios. Four concrete mixtures were prepared in this study, each with different percentages of metakaolin as partial substitution of

cement weights of 0%, 5%, 10%, and 15%. The water–binder ratio was fixed at 0.32 in all mixtures. The specimens underwent steam curing for 0, 4, 8, and 16 hours at a temperature of 55 °C.

2 Experimental study

2.1 Materials

The materials employed in this investigation include cement, water, aggregates, kaolin, and superplasticizer.

2.1.1 Cement

The study utilized the Blue Lion brand ordinary Portland cement manufactured by Cement Industries Malaysia Berhad. The cement complies with the ASTM C150/C150M-16 standard [27]. The cement has a specific gravity of 3.15.

2.1.2 Aggregates

The fine aggregates used were natural river sand, with a specific gravity of 2.75, a water absorption rate of 0.61%, and a fineness modulus of 3.10. These properties satisfied the standards set by ASTM C128-15 [28] and ASTM C136-06 [29]. The coarse aggregates consisted of crushed granite, with a maximum size of 12.5 mm. The bulk density of the coarse aggregates was approximately 1550 kg/m³, following the guidelines of ASTM C29/C29M-09 [30]. According to ASTM C127-15 [31], the specific gravity for the coarse aggregates was 2.70, with a corresponding water absorption of 0.49%. A sieve analysis was conducted on the fine and coarse aggregates to create gradation curves following the guidelines of ASTM C33/C33M-18 [32], as shown in Fig. 1.

2.1.3 Kaolin

The Kaolin used in the study was Refined Kaolin Akima 45, manufactured by Associated Kaolin Industries Berhad. The kaolin was packed in a 25-kg bag, as shown in Fig. 2. Table 1 illustrates the chemical components of ordinary Portland cement and metakaolin utilized in this investigation. Table 2 illustrates the physical features of metakaolin.

SiO₂ and Al₂O₃ are the main components of Kaolin clay, a white mineral primarily composed of hydrated aluminum di silicate (Al₂Si₂O₅(OH)₄), transforming into metakaolin crystal structure at 600 °C to 800 °C. Under-fired kaolin prevents amorphous phase conversion and pozzolanic formation, whereas over-fired kaolin results in dead burned mullite (3Al₂O₃·2SiO₂).

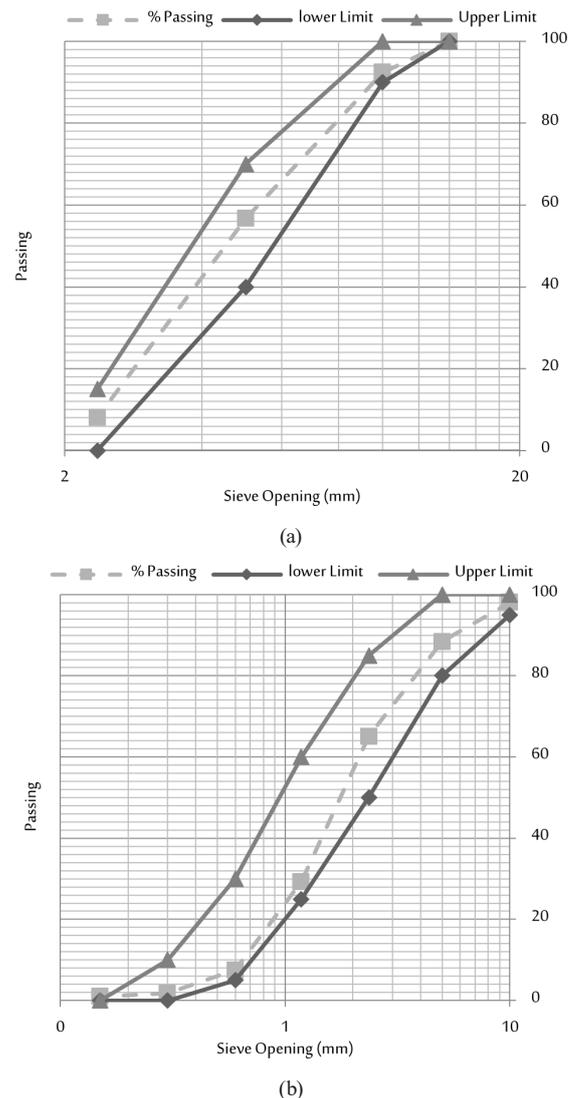


Fig. 1 Sieve analysis curves: (a) Coarse aggregate; (b) Fine aggregate

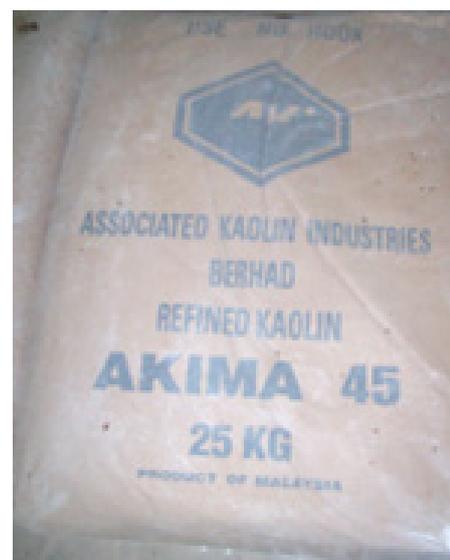


Fig. 2 Refined Kaolin Akima 45

Table 1 The chemical features of cement and metakaolin

Compositions	Cement	Metakaolin
SiO ₂	18.9	51.52
Al ₂ O ₃	4.9	40.18
Fe ₂ O ₃	3.8	1.23
CaO	65.77	2.0
MgO	0.81	0.12
K ₂ O	1.05	0.53
SO ₃	3.01	0.0
TiO ₂	0.26	2.27
Na ₂ O	0.07	0.08
LOI	2.52	2.01

Table 2 The physical features of metakaolin

Feature	Value
Specific gravity	2.6
Physical size	Powder
Color	Off-weight

2.1.4 Chemical admixture

The superplasticizer, with a trade name ADVA 181, was used as a chemical admixture during this study. This high-range retarding water-reducing admixture or superplasticizing admixture of type G utilizes the most recent comb polymer technology. The superplasticizer dosage amounts to 1.4% of the combined weight of cement and metakaolin.

2.2 Kaolin calcination

Kaolin was calcined by subjecting to a high temperature of 700 °C in a furnace, as shown in Fig. 3; it underwent three hours of heating. Subsequently, kaolin was allowed to cool to ambient temperature and stored in a plastic bag until the day of casting.

**Fig. 3** Calcination of kaolin in the furnace

2.3 Mixtures proportion

The investigation involved the preparation of concrete mixtures using a water–binder ratio of 0.32, aiming for a compressive strength of 60 MPa after 28 days. The tests were conducted using four specimen groups. The mixture proportions of the four specimens in the group are illustrated in Table 3. The first mixture served as the control (MK 0), consisting of 100% Portland cement (OPC), whereas the remaining mixtures incorporated different proportions of metakaolin in place of OPC. Table 3 displays the use of metakaolin at three different replacement ratios: 5% (MK 5), 10% (MK 10), and 15% (MK 15).

2.4 Specimen preparation

The mixing process began by adding the prescribed amounts of aggregates (i.e., sand and gravel) to the pan-type mixer. Subsequently, the binder (i.e., cement and metakaolin) was added to achieve complete mixing. The duration of this stage spans five minutes, allowing the aggregates and binder to be meticulously blended to attain a homogeneous dry mixture. Subsequently, the water and superplasticizer were incorporated into the mixture, and the mixing procedure persisted for approximately five minutes. The molds used to produce samples for compressive strength and initial surface absorption tests were cubes measuring 100 mm × 100 mm × 100 mm. Small cylindrical samples with 50 mm diameter and 40 mm thickness were used to determine water absorption and porosity. For the rapid chloride permeability test, a cylinder with 100-mm-diameter by 50-mm-thick samples was used. Methods of curing the concrete samples are shown in Table 4.

2.5 Curing

The control concrete specimens were subjected to moist curing in a fog room with high humidity at room temperature until they reached the testing ages of one, three, seven, and 28 days. The steam curing tank is shown in Fig. 4 (a). Immediately after casting, steam curing at atmospheric pressure was implemented for three different durations (excluding the delay period): four, eight, and 16 hours. To prevent microcracking due to variations in the coefficient of thermal expansion of the concrete components, the specimens were stored at room temperature (30 °C) for a specific amount of time prior to steam curing. The delay interval of 3 hours is selected due to its close approximation to the average setting time of the concrete. The peak temperature during the curing process reached 55 °C.

Table 3 Concrete mixture proportions

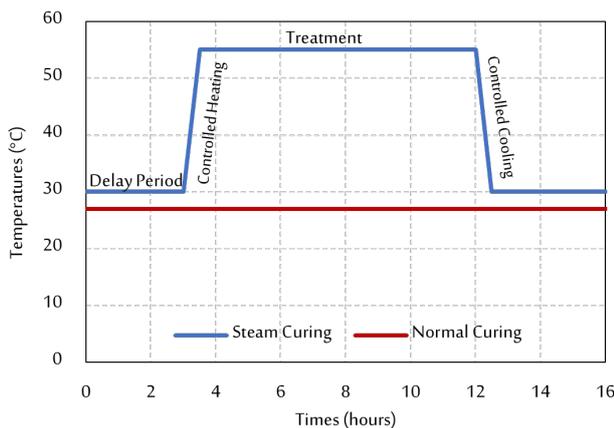
Mixture	Cement (kg/m ³)	Metakaolin (kg/m ³)	Fine aggregate (kg/m ³)	Coarse aggregate (kg/m ³)	Superplasticizer (L/m ³)	Water–binder ratio
MK 0	450	-	675	1118	6.3	0.32
MK 5	427.5	22.5	675	1118	6.3	0.32
MK 10	405	45	675	1118	6.3	0.32
MK 15	382.5	67.5	675	1118	6.3	0.32

Table 4 Methods of curing concrete samples

Test	Number of specimens											
	MK 0			MK 5			MK 10			MK 15		
	Steam curing (hours)			Normal curing			Steam curing (hours)			Normal curing		
Normal curing	4	8	16	4	8	16	4	8	16	4	8	16



(a)



(b)

Fig. 4 Steam curing: (a) Steam curing tank (b) Schematic process

A steam cycle involves a gradual increase in temperature up to a maximum of 55 °C over a duration of 30 minutes. Fig. 4 (b) displays the schematic representation of the steam curing technique. Subsequently, the curing process

involved steaming for durations of 4, 8, and 16 hours at a maximum temperature of 55 °C. A cooling period follows the steam curing stage at the prescribed temperature.

2.6 Test methods

2.6.1 Compressive strength test

The investigation involved measuring the compressive strength of three 100-mm cube specimens at various ages. The test was conducted at the ages of 1, 3, 7, and 28 days. A compression machine was used to apply the compression load at a rate of 0.3 N/mm²/s. Fig. 5 depicts the machine used for assessing the compressive strength. The test was carried out following the specifications outlined in BS EN 12390-3:2002 [33].

2.6.2 Rapid Chloride Permeability Test (RCPT)

The RCPT offers an indirect assessment of chloride permeability by tracking the amount of charge (in coulombs) that passes through a concrete sample. Two samples were tested for each mix at the ages of 3, 7, and 28 days. The equipment



Fig. 5 Compressive strength testing machine

utilized for the RCPT is depicted in Fig. 6. Cylindrical concrete samples with a diameter of 100 mm and thickness of 50 mm were extracted from 200 mm × 100 mm concrete cylinder according to AASHTO T277 [34]. The samples were placed into a vacuum desiccator for a duration of three hours. Subsequently, the samples were immersed in de-aired water, and then the pump was operated for an additional hour. Lastly, air was reintroduced into the desiccator, and the samples were immersed for 18 hours. Subsequently, the samples were inserted into the cell. The cathode of the cell is filled with a 3% sodium chloride (NaCl) solution. The positive side is immersed in a solution containing 0.3% sodium hydroxide (NaOH). A current of 60 volts DC was delivered to the ends of the samples for six hours. The total charge passed was determined and correlated with the resistance of the sample to the penetration of chloride ions.

2.6.3 Initial Surface Absorption Test (ISAT)

This test aims to achieve results that demonstrate the flow of water across a dry and level concrete surface. The test was conducted to obtain the duration required for a specific volume of water to pass through a glass tube placed on a concrete surface with a defined area. Fig. 7 displays

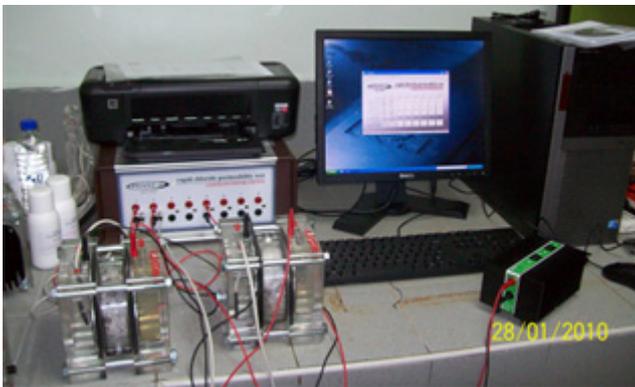


Fig. 6 RCPT equipment



Fig. 7 ISAT apparatus

the equipment used for ISAT. Prior to commencing the test, the equipment must be cleansed with a soap solution to reduce surface tension. Then, the tools are washed with distilled water. The vertical gap between the capillary and the horizontal concrete surface is consistently maintained at 200 mm, and the reservoir is filled with distilled water. Measurements are taken at intervals of 10 minutes, 30 minutes, 1 hour, and 2 hours while the water passes through the capillary, covering a distance of 100 divisions.

2.6.4 Porosity and water absorption test

A porosity test was conducted to evaluate the effect of metakaolin on the total porosity of concrete. The low value of the total porosity implies a potentially high durability and density of the concrete. A water absorption test was conducted to evaluate the water absorption ratio into the interconnected capillary pores in the concrete pore structure. Fig. 8 shows the specimens in the vacuum saturation apparatus. The specimens for these tests are 50 mm diameter × 40 mm thick cores extracted from a 100 mm × 100 mm × 500 mm beam and tested at the ages of 3, 7, and 28 days. The porosity and water absorption results were obtained using Eqs. (1) and (2), respectively, as follows:

$$\text{Porosity} = \frac{W_1 - W_2}{W_1 - W_3} * 100, \quad (1)$$

$$\text{Water absorption} = \frac{W_1 - W_2}{W_2} * 100, \quad (2)$$

where W_1 refers to the specimen's mass in a saturated and surface-dry state. W_2 refers to the specimen's mass after being oven-dried at a temperature of approximately 105 ± 5 °C for 24 hours. W_3 refers to the specimen's mass in water.



Fig. 8 Vacuum saturation equipment

2.6.5 Differential Thermal Analysis (DTA)

Differential thermal analysis (DTA) entails subjecting the test specimen and an inert reference to the same conditions of heating while simultaneously measuring any temperature discrepancy between the specimen and the reference. For this study, DTA was conducted using Shimadzu DTG 60 instrument. Cement pastes, with and without metakaolin, were mixed by hand for at least five minutes and cast in 35 mm × 25 mm plastic molds. The samples were steam-cured for 4, 8, 16, and 0 hours (control). The ages examined were 3, 7, and 28 days. Approximately 8–9 mg of a ground-hardened cement paste was added to the aluminum pan for DTA. Then, cement specimen is placed in the furnace and heated at a rate of 10 °C per minute until 600 °C was reached. Throughout the test, a 100-ml/minute nitrogen gas flow through the furnace maintained a controlled, inert nitrogen atmosphere. Fig. 9 shows the DTA instrument.

3 Results and discussion

3.1 Compressive strength

Fig. 10 represents the effect of steam curing on the compressive strength. The results of the compressive strength test show that the steam-cured concrete gains high strength at an early age, with or without metakaolin replacement, as shown in Fig. 10 (a). The compressive strengths of the concrete under normal curing were 19.24, 37.8, 35.1, and 35.6 MPa with 0%, 5%, 10%, and 15% replacement of metakaolin, respectively. The results indicate that the influence of steam curing is to enhance the one-day compressive strength of the concrete significantly comparison with the concrete cured under normal condition. During the four-hour steam curing period, the MK 5 (i.e., 5% replacement of metakaolin) had the highest compressive

strength of 49.9 MPa, which is 32% higher than normal curing, as shown in Fig. 10 (a). For eight and 16 hours of steam curing, 10% replacement (i.e., MK 10) showed the highest results with 52.7 and 57.28 MPa, respectively, 50% and 63% higher than normal curing. The results of compressive strength for three days are shown in Fig. 10 (b). At this time, metakaolin with 10% replacement (i.e., MK 10) has a higher compressive strength of 59.5 MPa after four hours and 59.9 MPa after eight hours of steam curing. However, for a 16-hour steam curing period, a 5% replacement level (i.e., MK 5) exhibited the highest compressive strength of 62.6 MPa, as shown in Fig. 10 (b). In terms of percentage increase, the concrete replaced with 5% metakaolin and steam cured for 16 hours had 18% higher compressive strength than the normally cured concrete, and that replaced with 10% metakaolin and steam cured for 16 hours exhibited a 10% compressive strength increase. Moreover, the concrete replaced with 15% metakaolin obtained a lower compressive strength than the 10% replacement level alone. This finding is consistent with the results of the study by Kocak [35], which indicated that increasing the replacement rate of cement weight with MK to 15% reduced the compressive strength of mortar samples at 2, 7, 28, and 56 days.

The compressive strength results for seven days of age are shown in Fig. 10 (c). At this age, the concrete replaced with 15% metakaolin has the highest compressive strength at a four-hour steam curing period with 66.17 MPa. For eight and 16 hours, replacement levels of 10% and 5% provide the highest compressive strength, with 65.15 and 69.33 MPa, respectively, as shown in Fig. 10 (c). For the 16-hour steam curing, MK 5 showed an increase of 23%, whereas MK 10 exhibited an increase of 12% compared with normal curing. At 28 days, the compressive strength development of the steam-cured concrete was slower than that of concrete subjected to normal curing, as shown in Fig. 10 (d). The concrete was replaced with 5%, 10%, and 15% metakaolin and steam-cured for 16 hours, showing a reduction in compressive strength of 1.2%, 10.1%, and 16.5%, respectively. This phenomenon can be attributed to the continuous formation of the C-S-H network in a high-temperature environment. This finding aligns with the increasing trend of compressive strength in high-strength concrete when combined with other admixtures under steam-curing conditions at early ages (from one to seven days) [36]. During the combined influence of heat and pressure, the pozzolanic activity experiences a rapid increase, leading to the filling of internal gel pores and



Fig. 9 DTA equipment

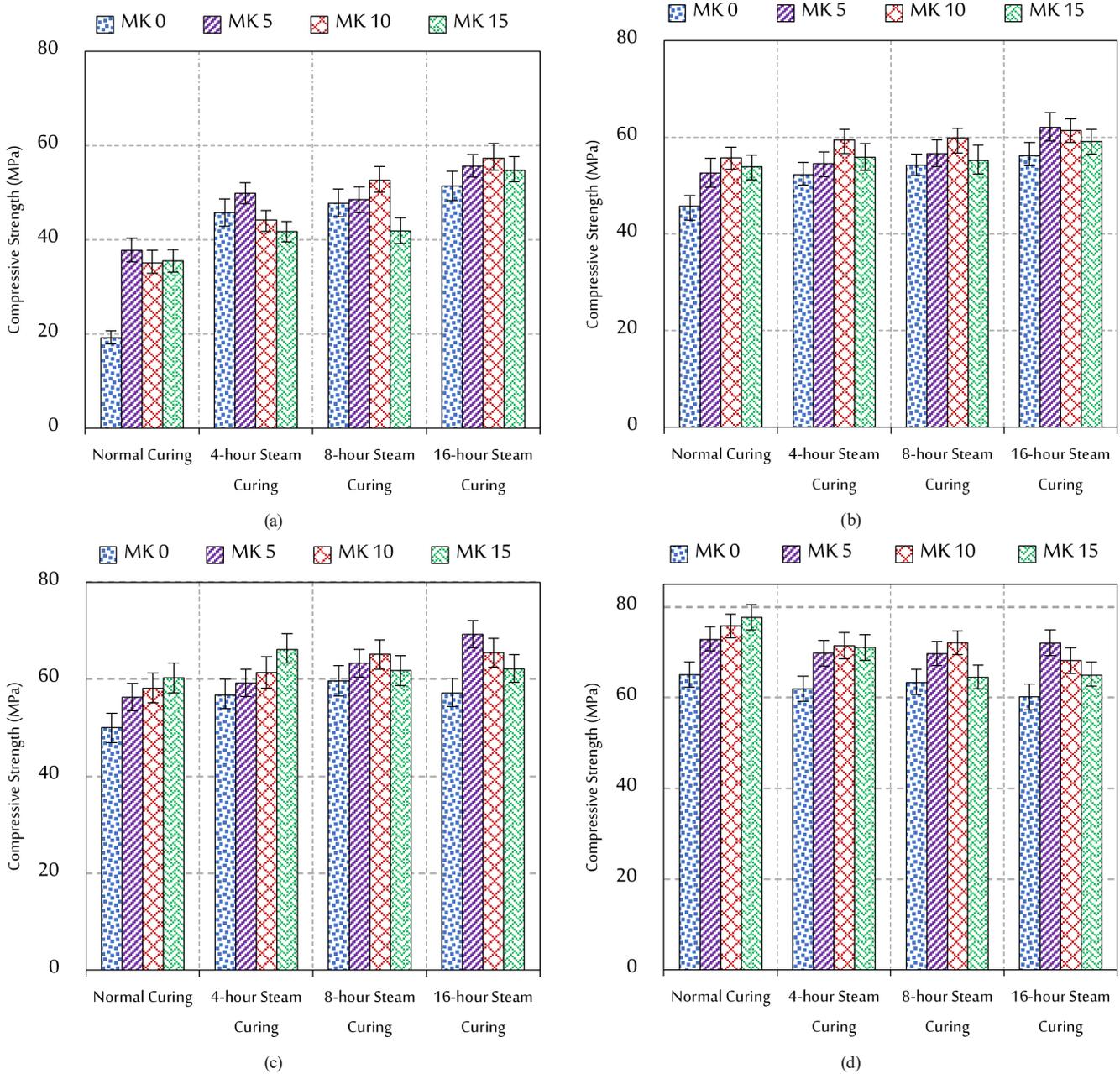


Fig. 10 Specimen's compressive strength at ages of: (a) 1 day; (b) 3 days; (c) 7 days; (d) 28 days

voids with hydration products [36]. Therefore, steam curing conditions can be employed to attain a high level of early strength. The decrease in compressive strength at 28 days with steam curing can be attributed to the coarsening of the rapidly developed microstructure, induced by the higher curing temperature [37].

3.2 Chloride permeability

The effects of the curing conditions and replacement level of metakaolin on rapid chloride permeability are depicted in Table 6 and Fig. 11. Metakaolin, when applied as a substitute for cement, and steam curing significantly reduced

the total charge passed compared with the control specimens, as evaluated by the RCPT. Fig. 11 (a) displays the results of a three-day rapid chloride permeability test. Fig. 11 illustrates that the concrete incorporating metakaolin and subjected to steam curing exhibited a significant reduction in the total charge passed. The total charge passed was reduced by increasing metakaolin replacement and the period of steam curing. Metakaolin with 10% replacement subjected to four-hour steam curing showed low total charge passed (<2000 *c*), whereas the eight-hour and 16-hour steam curing obtained extremely low (<1000 *c*) total charge passed. For the 15% replacement level of

Table 6 Total charge passed of all specimens

Mixture	Total charge passed (coulomb)											
	Normal curing			Four-hour steam curing			Eight-hour steam curing			16-hour steam curing		
	3	7	28	3	7	28	3	7	28	3	7	28
	days											
MK 0	3384	3367	1721	3141	2532	1471	2981	2282	1342	2718	1967	1276
MK 5	2718	1514	1398	2495	1417	1091	2165	1208	1065	2115	1127	931
MK 10	2318	684	572	1904	613	475	671	586	496	563	443	381
MK 15	949	386	354	860	356	227	613	302	257	430	284	256

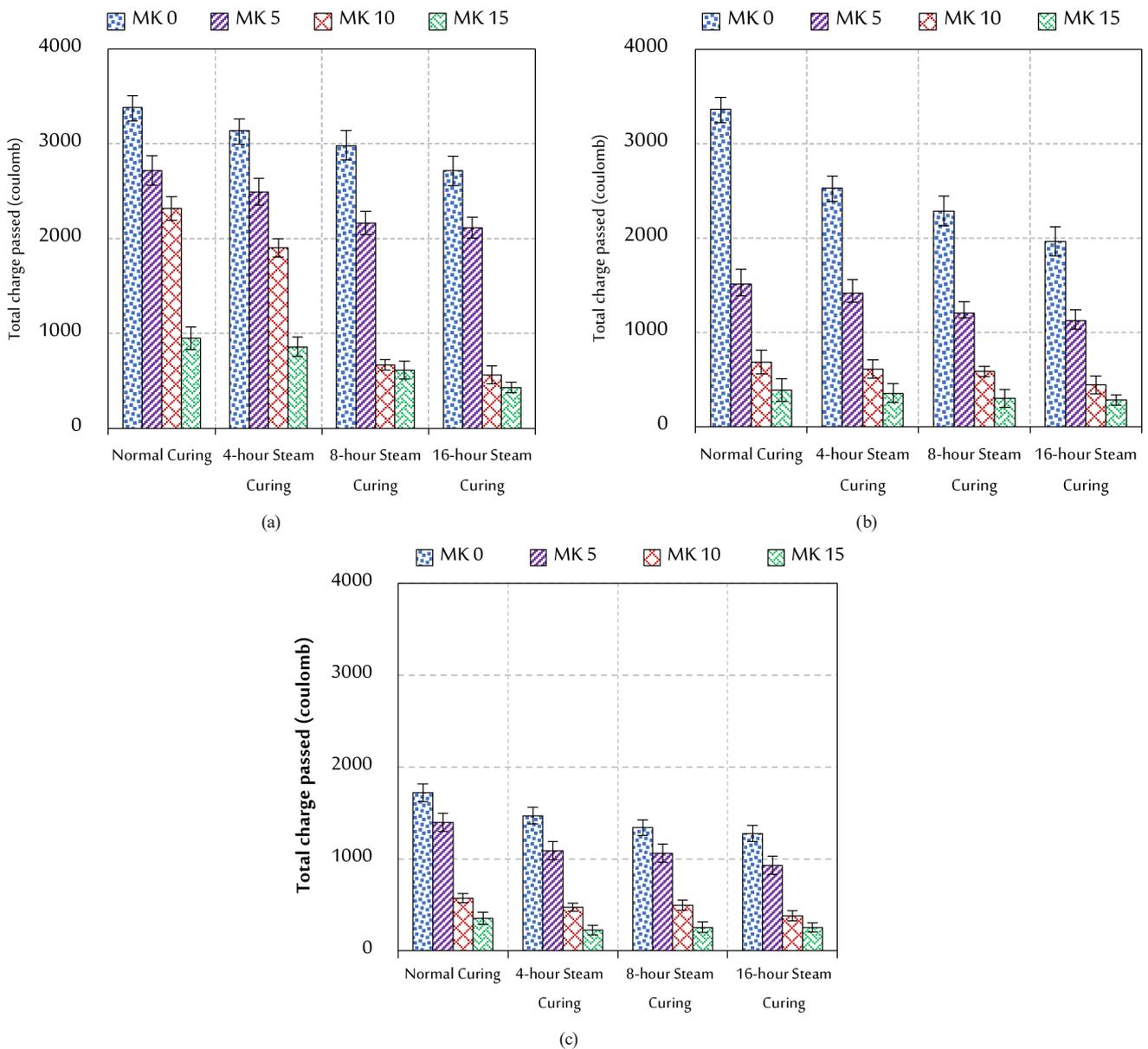


Fig. 11 Total charge passed of all specimens at the ages of: (a) 3 days; (b) 7 days; (c) 28 days

metakaolin, all curing periods proved to be effective in minimizing the total charge passed with a very small range (<1000 c). As shown in Fig. 11 (b), the seven-day results showed that 5% metakaolin replacement and steam-cured

concrete effectively reduced the total charge passed from moderate (>2000 c) to low (<2000 c). Concretes containing 10% and 15% metakaolin exhibit a significant decrease in the total charge passed with a very low value (<1000 c)

for all periods of steam curing. At this age, concrete with 15% replacement and steam-cured for 16 hours obtained lower total charge passed of 264 *c*. The results at 28 days of age are illustrated in Fig. 11 (c), illustrating a reduction in the total charge passed. The 5% metakaolin replacement and steam curing showed low (<2000 *c*) total charge passed, whereas the 10% and 15% replacement obtained an extremely low (<1000 *c*) total charge passed at all steam curing periods. The concrete with 15% replacement and steam curing for four hours showed the lowest total charge passed with 227 *c*. The results of the RCPT indicate that using metakaolin as a partial substitute material notably enhanced the concrete's resistance to chloride ion penetration. The findings are consistent with the results reported in the literature [38]. This phenomenon is anticipated because the presence of ultrafine particles enables mineral admixtures to move into the voids in the cement paste matrix and the transition zones between the cement paste matrix and aggregate particles [39]. As a result, a denser microstructure is formed. The penetration rate of chloride into concrete is influenced by the concrete's pore structure, which is influenced by several factors, such as materials, construction techniques, and age. A low water–binder ratio (which in this study was 0.32) [40], the use of metakaolin [41], and the use of steam curing [37] to speed up the concrete's hydration process contribute to the low rate of chloride ion migration as reflected by the low total charge passed.

3.3 Initial surface absorption

The ISAT measures the rate at which water flows into the concrete's surface per unit area. The results of the ISAT are presented in Table 7 and Fig. 12. Fig. 12 illustrates that the absorption of water through the surface of concrete containing metakaolin reduces with age for the normal and steam curing. When normal curing is implemented on the concrete, the replacement of metakaolin can reduce the surface absorption. As the replacement level increases, the surface absorption decreases. As shown

in Table 7, 15% replacement of metakaolin in concrete exhibited the lowest absorption value, regardless of the steam curing period. Moreover, the initial surface absorption decreased with an increasing steam curing period. At three days, 10% replacement with 16-hour steam curing provides a 36% reduction in the initial surface absorption compared with the normally cured concrete. For seven days, the results show that 5% replacement with a 16-hour steam curing exhibits a 22% reduction in the initial surface absorption. At 28 days, the concrete with 10% replacement of cement by metakaolin with 16 hours of steam curing records a 29% reduction in initial surface absorption compared with normally cured concrete. This phenomenon is acceptable because the porosity of the concrete reduces with age as hydration products fill the pores that exist in the concrete. Given that the hydration rate is higher at an early age, the ability to absorb water reduces rapidly at this moment. On the contrary, when the hydration rate decreases at a later age, the reduction in water absorption on the concrete surface is not evident. This finding can be attributed to the fact that when concrete is exposed to high temperatures, an increase in the metakaolin replacement level reduces the porosity. This statement is supported by the experiment carried out by Poon et al. [42] to study the performance of concrete containing metakaolin at elevated temperatures. When the metakaolin replacement in the concrete increases, an increase in exposure temperature up to 60 °C decreases the concrete porosity. As a result, surface absorption decreases with an increase in metakaolin replacement level under steam curing. This finding also conforms to the research of Rojas and Sánchez de Rojas [43], indicating that the average pore diameter reduces with an increase in metakaolin replacement level under steam curing. The same observations are found for other metakaolin concrete, where steam curing can reduce their water absorption ability in the presence of metakaolin. However, for concrete without metakaolin, the initial surface absorption increases with the increase in the period of

Table 7 Initial surface absorption of all specimens

Mixture	Initial surface absorption (ml/m ² /s)											
	Normal curing			Four-hour steam curing			Eight-hour steam curing			16-hour steam curing		
	3	7	28	3	7	28	3	7	28	3	7	28
MK 0	0.0348	0.0248	0.0241	0.0357	0.0268	0.0249	0.0377	0.0287	0.0251	0.0416	0.0288	0.0261
MK 5	0.033	0.0287	0.0233	0.0285	0.0273	0.0238	0.0254	0.0243	0.0226	0.0227	0.0223	0.0215
MK 10	0.0278	0.0204	0.0179	0.0238	0.017	0.0176	0.0198	0.0167	0.0168	0.0176	0.0163	0.0125
MK 15	0.0184	0.0185	0.0147	0.0178	0.0165	0.0097	0.0169	0.0159	0.0104	0.018	0.0154	0.0121

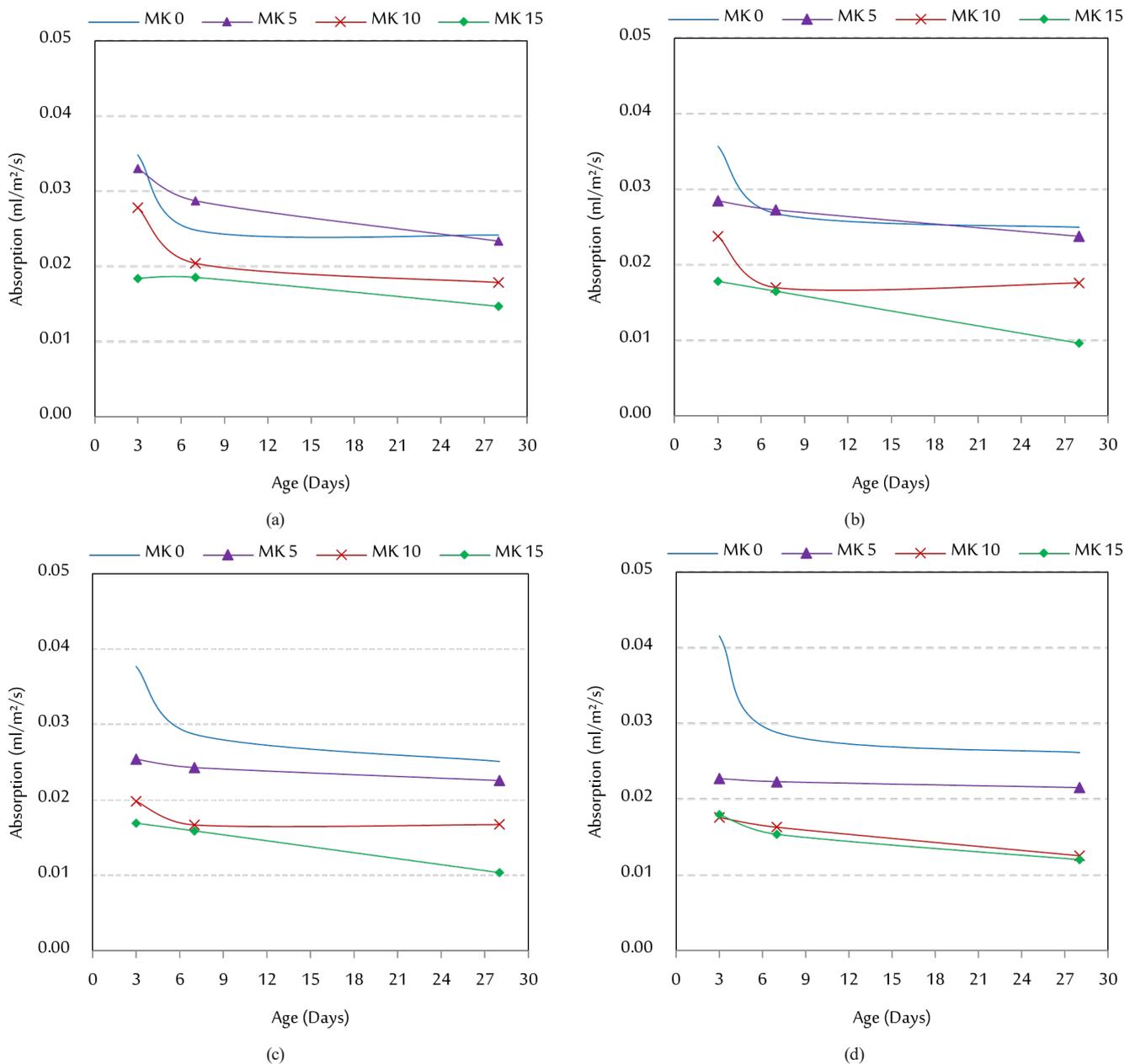


Fig. 12 Initial surface absorption of all specimens: (a) Normal curing; (b) 4-hour steam curing; (c) 8-hour steam curing; (d) 16-hour steam curing

steam curing. Control specimens (i.e., MK 0) treated with steam curing led to the formation of a significant capillary porosity. Hydration, which achieves uniform pore-filling and decreases the porosity, is inconsistent. This situation leads to increased porosity in the concrete. Ho et al. [44] and Campbell and Detwiler [45] also reported a comparable increase in water absorption during steam curing.

3.4 Water absorption

The water absorption test was carried out using the water immersion under vacuum method to calculate the amount of pore space in concrete that fluids can enter. The water absorption results are shown in Table 8 and Fig. 13. As shown in Fig. 13, for all curing conditions, the water

absorption reduces with age for all concretes with or without metakaolin. As shown in Table 8, when normal curing (control) is implemented, water absorption decreases with the increase in metakaolin percentage. When subjected to a steam curing period, water absorption increases with the increase in the steam curing period. For the three-day curing results, the 5% replacement level and 16-hour steam-cured concrete showed the highest water absorption, increasing by 47% compared with the normal-cured concrete. The seven-day curing results show that the 15% replacement level subjected to steam curing for eight hours had the highest increase of 65% water absorption. For the 28-day curing results, the 10% replacement level and 16-hour steam-cured concrete increased the water

Table 8 Water absorption of all specimens

Mixture	Water absorption (%)											
	Normal curing			Four-hour steam curing			Eight-hour steam curing			16-hour steam curing		
	3	7	28	3	7	28	3	7	28	3	7	28
MK 0	3.178	3.219	2.423	3.312	3.225	2.502	4.402	3.762	2.688	4.657	4.119	2.777
MK 5	3.05	3.141	2.231	3.162	3.067	2.350	4.37	3.553	2.555	4.487	3.877	2.630
MK 10	2.727	2.406	2.063	2.976	2.687	2.291	3.229	3.116	2.442	3.466	2.921	2.529
MK 15	2.434	1.766	1.945	2.313	2.351	2.309	3.152	2.931	2.047	2.901	2.727	2.061

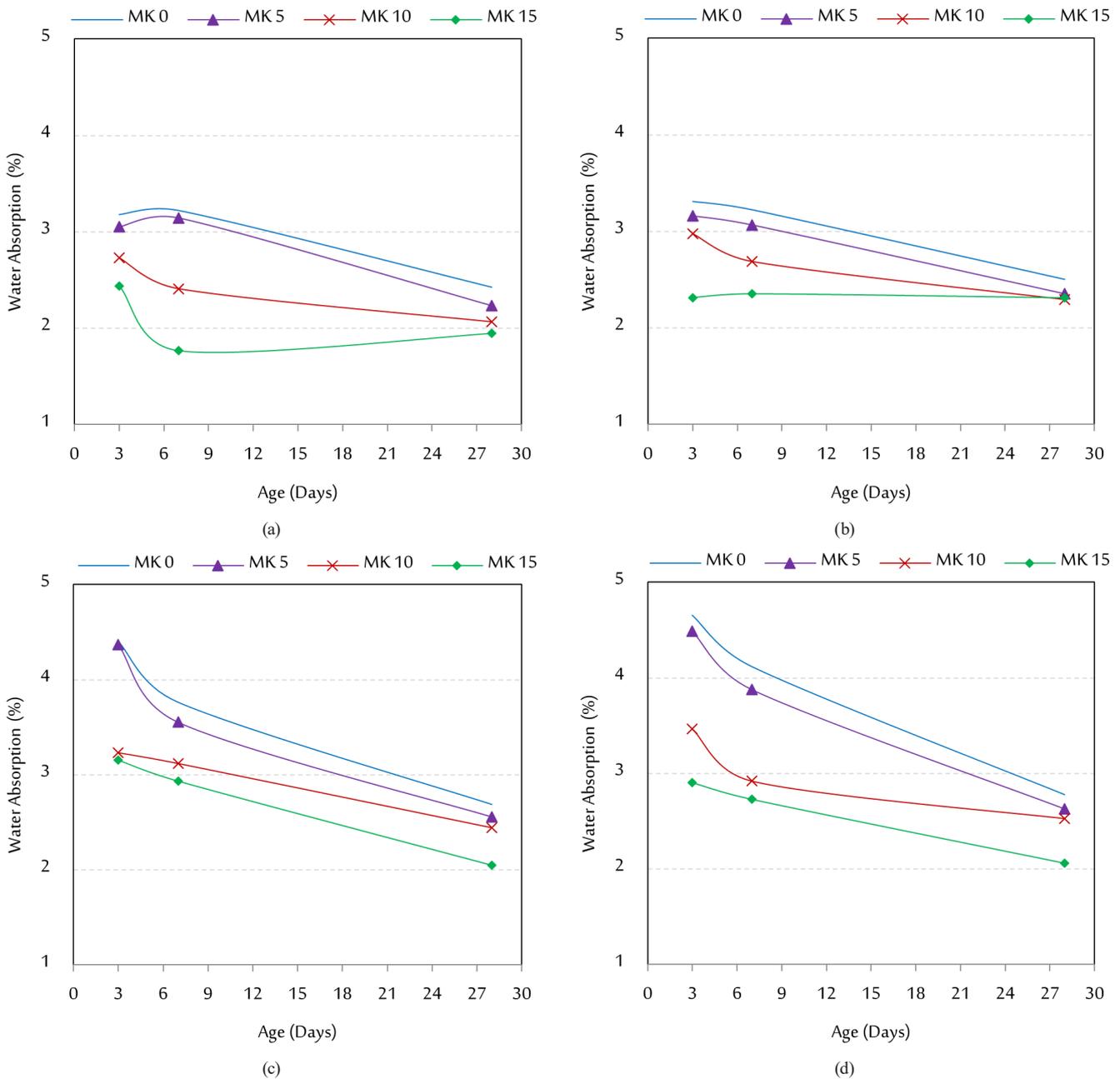


Fig. 13 Water absorption of all specimens: (a) Normal curing; (b) 4-hour steam curing; (c) 8-hour steam curing; (d) 16-hour steam curing

absorption by 22% compared with the normal-cured concrete. This phenomenon occurs due to the continuous hydration process, resulting in the development of

concrete with reduced porosity. As the hydration process occurs, the resulting products fill the pores in the hardened cement paste phase of the concrete, decreasing the

average size of the pores. Consequently, the concrete's capacity to absorb water diminishes. According to Khatib and Clay [46], absorption mainly depends on the total pore volume. Based on Bredy et al.'s [47] research, when the metakaolin level was less than 20%, the overall pore volume of the paste was reduced. When steam curing is implemented, water absorption decreases with increasing percentage replacement of metakaolin. Rojas and Sánchez de Rojas [43] found that a cement paste containing metakaolin, when cured at 60 °C, reduces the pore size.

3.5 Differential thermal analysis

DTA identifies the temperature ranges at which the various phases in the cement paste undergo thermal decomposition. DTA is a valuable tool for monitoring changes in hydration and assessing the progress of pozzolanic reactions, including the depletion of calcium hydroxide (Portlandite). CH is formed as a result of the hydration of C_2S and C_3S . Contrary to C-S-H, CH does not play a significant role in enhancing the strength of concrete and can harm its durability. Moreover, CH takes up a significant amount of space in the cement paste and has a tendency to expand into any available voids until it is prevented [48]. Upon the addition of metakaolin, it exhibits a rapid reaction with the CH, thereby producing secondary C-S-H. Metakaolin results in the formation of several calcium aluminate compounds.

In addition, the use of metakaolin as a partial cement substitute enhances the formation of cement hydration products as a result of the pozzolanic reaction with CH. Thus, pozzolanic reaction processes consume CH and form additional gels of calcium aluminosilicate hydrate (C-A-S-H) in addition to calcium silicate hydrate (C-S-H). This phenomenon is due to the addition of metakaolin, which contains a high percentage of aluminum that changes and enhances the gel content during the pozzolanic reaction. This finding is demonstrated through the use of DTG analysis and other tests, which yield positive results in terms of strength, transport, and microstructural properties [49].

Decreasing the amount of CH is advantageous because it produces additional C-S-H and reduces the potential leaching of CH [50]. Hence, measuring the amount of CH present in a sample provides insight into the advancement of the pozzolanic reaction process of metakaolin. The typical thermal decompositions of several phases in cement paste were mentioned in previous studies [51]. DTA was conducted on cement pastes containing varying percentages of metakaolin (0%, 5%, 10%, and 15%) that were subjected to steam curing for different durations (4, 8, and 16 hours) as well as conventional curing. The analyzed

results are depicted in Fig. 14. The DTA curves show that the hydrates present in the paste are C-S-H gel and CH (Portlandite). As shown in Fig. 14 (a), at three days, in normal curing (control) cement paste, the endothermic peaks for 0%, 5%, 10%, and 15% are similar. The decomposition temperature for CH is between 380 °C and 450 °C. In cement paste with higher replacement containing 15% metakaolin, CH decomposition begins early. A weak endothermic occurs at temperatures between 120 °C and 140 °C, which is attributed to C-S-H. Therefore, the pozzolanic reaction that occurs between metakaolin and CH has already started [52]. The relative quantities of CH can be ascertained by comparing DTA peak parameters, such as area and amplitude. For three days, as shown in Fig. 14 (a), 16-hour steam-cured metakaolin cement paste showed lower CH content in comparison to other three-day curing conditions. This finding is due to the high pozzolanic reaction that occurs between metakaolin and CH. Cement paste containing 15% metakaolin shows a remarkably less pronounced peak at temperatures between 395 °C and 427 °C. The C-S-H endothermic peak occurs at 144 °C. The DTA results for seven days and 28 days are shown in Fig. 14 (b), (c). The 16-hour steam-cured cement paste with a replacement of 15% metakaolin also has a lower CH content because of the high pozzolanic reaction. The addition of metakaolin during steam curing decreases the CH concentration in the cement paste.

4 Conclusions

Based on the experimental findings of this study, the following conclusions are drawn:

1. The compressive strength of concrete with metakaolin as cement partial replacement increased with steam curing, with higher results at an early age. At three days, the strength of MK 5 concrete with 16-hour steam curing increased by 18% compared with normally cured concrete. However, the 28-day compressive strength is lower than that of the normally cured concrete.
2. The rapid chloride permeability test results indicate that metakaolin partial replacement and steam curing improve concrete resistance against chloride penetration. The total charge passed is reduced by increasing the metakaolin replacement and the period of steam curing.
3. Initial surface absorption decreases with increases in metakaolin replacement level when normal and steam curing are implemented. The MK 15 exhibited the lowest water absorption value, regardless of the steam curing period.

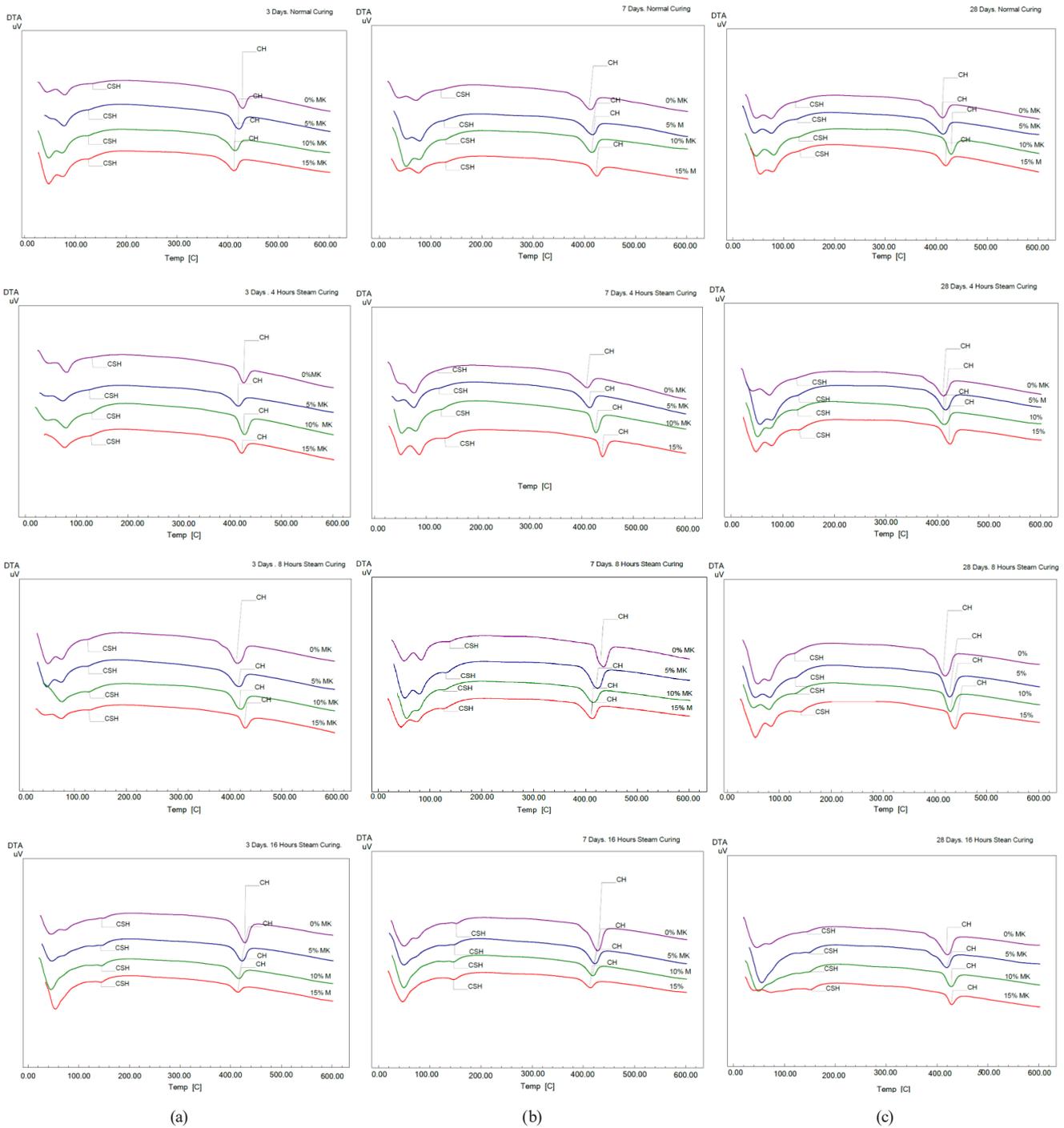


Fig. 14 DTA curves for cement pastes with 0%, 5%, 10%, 15% metakaolin at age of: (a) 3 days; (b) 7 days; (c) 28 days

4. Water absorption decreases with metakaolin replacement in normal curing, but it increases with an increase in the steam curing period for concrete with and without metakaolin. At seven days, the absorption of MK 15 concrete with eight hours of steam curing increased by 65% compared with the normally cured concrete.
5. Differential thermal analysis shows that a high pozzolanic reaction between metakaolin and CH occurs with high replacement metakaolin (15%) and longer steam curing period (16 hours).
6. Steam curing significantly enhances the performance of high-strength concrete at early ages of three days by stimulating reaction processes.
7. Therefore, the best replacement of cement with metakaolin ranges between 10% and 15% of the cement weight to achieve superior strength and transport properties of high-strength concrete when applying steam curing regimes.

5 Future investigations

In order to expand the scope of research in the field of the effect of steam curing on the performance of high-strength concrete containing metakaolin, the following future study suggestions can be used:

1. Examine the effect of processing temperatures at 65 °C, 75 °C, and 85 °C as a continuation of ongoing research.
2. Evaluate the effect of steam treatment duration exceeding 24 hours on microstructural properties.
3. Assess the effect of a high replacement rate of more than 15% by weight of cement on HSC properties. Adding other pozzolanic materials besides metakaolin is recommended.
4. Study and explore the potential economic and environmental benefits of incorporating metakaolin into concrete mixtures for steam curing applications.
5. Design experiments to improve the metakaolin replacement ratio in cement to achieve the best balance between strength enhancement and transport properties.

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Abbreviations

GHGs	: Greenhouse gases
CO ₂	: Carbon dioxide
CH	: Calcium hydroxide
C-A-S-H	: Calcium aluminosilicate hydrate
C-S-H	: Calcium silicate hydrate
ASTM	: American society for testing and materials
OPC	: Portland cement
RCPT	: Rapid chloride permeability test
NaCl	: Sodium chloride
NaOH	: Sodium hydroxide
ISAT	: Initial surface absorption test
DTA	: Differential thermal analysis
C ₂ S	: Dicalcium silicate
C ₃ S	: Tricalcium silicate

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