



Research Article

Effects of reactive MgO and metakaolin on compressive strength and chloride migration coefficient of sodium carbonate activated slag mortars

Mehrnosh ABOLFATHI¹, Serhan ULUKAYA¹, Büşra AKTÜRK^{2*}

¹Department of Civil Engineering, Yıldız Technical University, Istanbul, 34349, Türkiye

²Department of Civil Engineering, Istanbul Bilgi University, Istanbul, 34060, Türkiye

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ABSTRACT

This paper presents an investigation of the compressive strength and chloride migration of sodium carbonate (NC) activated slag-based mortars. In the experimental study, NC was preferred to use as an activator since it can be designated as an environment-friendly activator. To improve the performance of sodium carbonate-activated slag-based mortars (NCAS), reactive MgO (M) and metakaolin (MK) were replaced with slag up to 15% by mass and five mortar mixtures were prepared with different M and MK replacement ratios. It was found that replacing the slag with a combination of 10% M and 5% MK achieved the highest compressive strength for both early age and ultimate strength. In addition, the mix which was composed of 85% slag, 10% M and 5% MK, was the most resistant one against chloride migration. Based on the compressive strength and chloride migration test results, it was concluded that the compressive strength and chloride migration performances of NCAS could be enhanced by the combination of M and MK.

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INTRODUCTION

In recent years, emphasis has been placed on using environment-friendly materials in concrete production to reduce CO₂ emissions. One of the studies under this topic is the use of aluminosilicate raw materials activated with any alkaline reagent instead of cement. These materials

are called geopolymer or, in general terms, alkali-activated materials (AAM) [1]. In this context, the main goal of the studies is to produce sustainable binders with the use of waste materials in the concrete industry and improve their mechanical properties and durability. On the other hand,

*Corresponding author.

*E-mail address: bucarkosar@gmail.com; mehrnoshabolf@gmail.com, serhanu@yildiz.edu.tr

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the cost required for cement production and disposal of by-products is taking up remarkable effort [2,3].

Alkali-activated binders are divided into two models, as Chao Li et al. mentioned [4]. One is the calcium-enriched systems, and the other one is alumino silicate-based systems. This study presents the former model binder activated with sodium carbonate.

Many studies focused on the activator type and dosage, internal structure, and glassy phase amount of alkali-activated slag (AAS) based systems. They provided extensive information about the mechanical and durability properties of AAS [5-8]. However, most of these studies emphasize the use of sodium hydroxide (NH) and sodium silicate (NS) as an activator. In this study, sodium carbonate (NC) was preferred to use instead of NH and NS. NC can be defined as environment-friendly material since it exists in nature as a trona ore mineral, thus causing a lower carbon footprint. Since NC only produces carbonate ions during the dissolving process in water, it is known as less caustic and less harmful than other commonly used activators. Moreover, NC is several times cheaper than NH or NS. [9]. On the other hand, delayed setting time and strength development are the major disadvantages of NC-activated systems [10-13]. Researchers reported different arguments about how the reaction mechanism proceeds in NC-activated slag systems. The reason for the delayed reaction mechanism of NC activated slag systems was reported by Jimenez and Puertas by the presence of calcium carbonate and mixed sodium/calcium carbonate double salts [11]. On the other hand, Bernal et al. [10] demonstrated the CO_3^{2-} ion concentration in the pore solution and the initially precipitated calcium carbonate control the NC activation process to a considerable extent. The delayed hardening process in NC-activated materials is due to the slow development of alkalinity required to initiate dissolution of the slag. It is known that initially, Ca^{2+} ions from slag react with CO_3^{2-} ions sourced by the activator and form carbonate salts such as calcite (CaCO_3) and gaylussite ($\text{Na}_2\text{Ca}(\text{CO}_3)_2 \cdot 5\text{H}_2\text{O}$). Accordingly, the pH of the medium increase through the consumption of CO_3^{2-} ions. These appear before the C-(A)-S-H gel precipitation. The pH value increases slowly when there is an excess of CO_3^{2-} ions in the system. When CO_3^{2-} ions are exhausted, the next stage of the reaction is comparable to NH-activated slag [14].

NC-activated slag systems (NCAS) have comparable compressive strength with OPC due to the various carbonate-containing phases. The primary hydration products are transit phases like gaylussite and calcite. The main binder phases of NCAS have been described as calcium aluminum silicate hydrate (C-A-S-H) and hydrotalcite. [15-17]. On the other hand, systems containing alumino-silicate resources such as metakaolin (MK) forms sodium aluminum silicate hydrate (N-A-S-H) gels as hydration products, contributing to strength-giving phases [4,18].

Incorporating MK in the NC-activated slag systems may enhance the compressive strength due to the effect of MK by increasing dissolved Al^{3+} and Na^+ ions in the system. These dissolved ions lead to the formation of C-S-H gel and Al or Na-enriched phases [19]. On the other hand, reactive MgO produced by the calcination of magnesite at relatively low temperatures (650–800 °C) might be another suitable additive to enhance the reaction mechanisms of NCAS. MgO dissolves in the water and creates an alkaline environment, leading to the breakage of Si-O-Si and Al-O-Si bonds of slag. Then, M ions react with broken Si-O and Al-O bonds and form hydrotalcite and magnesium silicate hydrate (M-S-H) which are described as strength-giving phases [18].

Chloride penetration is one of the important durability parameters affecting reinforced concrete's long-term performance, which depends mainly on the microstructure, connectivity, and tortuosity of the pore structure [20]. Most of the AAMs show high resistance to chemical attacks such as chloride penetration [21,22] due to their low permeability. Bondar et al. [23] investigated the chloride diffusivity of NH and NS-activated slag concrete with various alkali concentrations and silicate modulus. They reported that the chloride diffusion coefficient was still lower than the OPC concrete despite the higher water to binder ratio. Liu et al. [24] investigated the chloride resistance of alkali-activated fly ash slag blends containing MgO. They found that MgO improved the chloride resistance of the alkali-activated fly ash slag matrix. Vo et al. [25] studied the effects of fly ash and reactive MgO on sodium silicate-activated slag-based concretes. The authors indicated that incorporating reactive MgO up to 5% by weight was most successful in improving chloride penetration resistance in the concrete samples since MgO accelerated the hydration reaction of AAS and produced hydration products hydrotalcite-like phases to fill the pores.

In this study, the compressive strength and chloride migration coefficient of NCAS are investigated. Reactive MgO and MK are replaced with slag to research for possible enhancement of the performance of the mixes. It was obtained that any study about the compressive strength and chloride migration performance of NCAS systems containing a combination of reactive MgO and MK did not report yet. It was found that using a mix of metakaolin and reactive MgO can enhance both compressive strength and chloride migration resistance of NCAS systems. The outcomes of this study may help the designers to produce a new, sustainable, and high-performance AAS system.

MATERIALS AND METHODS

Materials, Mix Design, and Sample Preparation

The ground granulated blast furnace slag was used as the main binder and provided by Eregli Iron and Steel Factory. The other raw materials, reactive MgO and metakaolin,

were obtained from KUMAS Magnesia and Kaolin EAD. The chemical compositions of the raw materials were determined by X-ray fluorescence and presented in Table 1.

The particle size distributions of slag, reactive MgO (M), and metakaolin (MK) were obtained by Mastersizer. The particle sizes range from 0.3 to 105 μm with a d50 of 10 μm for slag, range from 0.4 to 315 μm with a d50 of 26 μm for M and range from 0.4 to 91 μm with a d50 of 4 μm for MK. As a fine aggregate, siliceous sand with a specific gravity of 2.67 was used and its ratio in the produced mortars was kept constant at 50% by volume. The particle size distributions and physical properties of these constituent materials are given in Figure 1 and Table 1.

A total of five mortars were produced to determine the compressive strength and chloride migration coefficient of NCAS mixes. Sodium carbonate (Na_2CO_3) with a specific gravity of 2.54 was used as an activator. In the mixtures, slag was replaced by M and MK up to 15% by mass, and Table 2

presents the proportions of the mortar mixes. The control mix in which the slag was solely used as a binder was coded as S, and the other four mixes were coded depending on the M and MK replacement ratios.

NC was used at 10% by mass of the total binder and was dissolved with tap water to prepare the activator solution. The water to binder ratio (w/b) by mass was 0.45 for all samples. After the mixing, the fresh mortar mixes were cast into cubic molds with dimensions of 50 \times 50 \times 50 mm and cylindrical molds with dimensions of ϕ 100/200 mm for compressive strength test and chloride migration test, respectively. To prevent moisture losses during the curing time, the molds were covered with plastic sheets. During the first 24 hours, the specimens were kept in ambient conditions and then stored in an oven with a temperature of 60°C for 24 hours. At the end of 48 hours, the specimens were demolded and kept under ambient conditions with a temperature of 20 \pm 2°C and relative humidity of 55 \pm 5% until the test day.

Table 1. Chemical composition and physical properties of raw materials

Chemical composition (%) by wt.	Slag	MgO	Metakaolin
SiO_2	36.6	6.0	56.1
Al_2O_3	14.2	0.7	40.25
Fe_2O_3	1.0	0.3	0.85
CaO	34.8	3.3	0.19
MgO	8.3	83.6	0.16
SO_3	0.86	0.2	–
Cl	0.015	0	–
Na_2O	0.42	0.4	0.24
K_2O	0.74	0	0.55
TiO_2	1.04	–	0.55
Mn_2O_3	2.29	–	–
LOI (%)	–	–	1.11
Specific surface area (m^2/g)	0.51	1.87	3.19
Specific gravity	2.93	3.55	2.30

Table 2. Mix design of mortar samples

Mix	Precursor (% mass)			W/B	Mixture composition (kg/m^3)					
	Slag	M	MK		Slag	M	MK	Water	NC	Sand
S	100	0	0		632	0	0	284	63	1335
5M10MK	85	5	10		537	32	63			
10M5MK	85	10	5	0.45	537	63	32			
15M	85	15	0		537	95	0			
15MK	85	0	15		537	0	95			

TEST METHODS

Compressive Strength Development

The compressive strength of the mortars was determined according to the ASTM C109 [26]. The compression test was conducted on triplicate specimens for

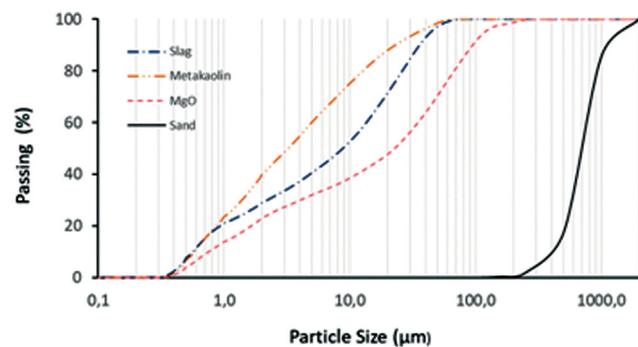


Figure 1. Particle size distribution of slag, metakaolin, reactive MgO, and sand.



Figure 2. The set up for non-steady state chloride migration test.

each mortar type at 3, 7, 28, and 90 days with a loading rate of 1.5 kN/s.

Chloride Migration Coefficient

In this study, NT BUILD 492 standard [27] was used to measure the resistance of the mortar specimens to chloride penetration. This method requires the cylindrical specimens to be cut into 5 cm slices. After brushing and cleaning the surfaces from any burrs, first, dry specimens were placed in a vacuum container and absolute pressure was set at 0.5 bar. This treatment was maintained for three hours. Subsequently, while the vacuum pump was still running, the container was filled with a previously prepared solution which was saturated with calcium hydroxide $[\text{Ca}(\text{OH})_2]$ by using distilled water. These samples were kept in limewater under vacuum for 18 ± 2 hours.

After the catholyte and anolyte solutions were prepared, the lateral surfaces of the samples were wrapped with ethylene propylene diene monomer tape and then fitted into a rubber sleeve to expose to NaCl solution only from one side. The purpose of this process is to avoid mixing cathodic and anodic waters. The test period was 24 hours and the appropriate voltage range (U) was applied according to the initial current (Fig. 2). At the end of the test, the samples were split into two and a silver nitrate (AgNO_3) solution was immediately sprayed to determine the chloride penetration depth [28]. The non-steady-state migration coefficients were calculated on two samples for each mix by Eq. (1-3).

$$D_{nssm} = \frac{RT}{zFE} \times \frac{X_d - \alpha\sqrt{X_d}}{t} \quad (1)$$

$$\alpha = 2\sqrt{\frac{RT}{zFE}} \times \text{erf}^{-1} \left(1 - \frac{2C_d}{C_0} \right) \quad (2)$$

$$E = \frac{U-2}{L} \quad (3)$$

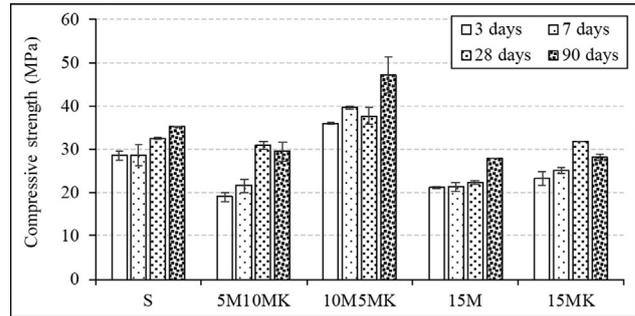


Figure 3. Compressive strength values of mortars at different ages.

where, D_{nssm} is non-steady state chloride migration coefficient (m^2/s), $z = 1$, $R = 8.314 \text{ J}/(\text{K}\cdot\text{mol})$, $F = 9.648 \times 10^4 \text{ J}/(\text{V}\cdot\text{mol})$, T is the average temperature of NaOH solution (K), X_d is the average of the penetration depths (m), t is the test duration (s), the c_d is the chloride concentration which equals to 0.07 N, and c_0 is the chloride concentration of NaCl solution which equals to 2 N.

In Eq. (3), L is the thickness of the specimen (m), and U is the applied voltage (V). According to this simplification, each mixture's chloride migration coefficient was calculated by taking the average of the two samples into account.

RESULTS AND DISCUSSION

Compressive Strength

The strength development of NCAS mortars are shown in Figure 3. The 3, 7, and 90 days compressive strength of mixes ranged from 19.0 to 36.0 MPa, 21.4 to 39.1 MPa, 22.3 to 39.6 MPa, and 27.9 to 47.2 MPa, respectively. The lowest and highest strength values at early ages 3 and 7 days belonged to 5M10MK and 10M5MK mixes. However, this trend changed for 28 days compressive strength; the lowest value was obtained in 15M mixes, while 10M5MK had the highest value. For the long-term period of curing, for 90 days, 10M5MK mixes reached the highest value with 47.2 MPa. It can be seen that replacing slag with only M or MK resulted in lower compressive strength values compared to the S mortar. For both 15M and 15MK mixes, the decrease percentages in the compressive strength were similar. Only 10M5MK exceeded the strength values of S mixes at all ages. 10M5MK mortar presented the highest compressive strength at all ages (Fig. 3).

Using a low amount of MK (5%) and a moderate amount of M (10%) significantly increased the compressive strength values. Compared to S mixes, these increment values were 26% and 34% for 3 and 90 days, respectively. On the other hand, MK incorporated mixes reached higher strength values over time. 5M10MK strength value increased

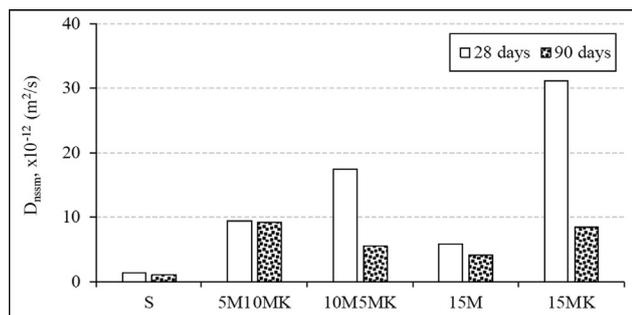


Figure 4. Chloride migration coefficients of mortars.

approximately 63% from 3 days to 90 days, while this value was 24% for S mixes.

NC, as an activator, has not been studied widely in alkali-activated systems due to its drawbacks, such as relatively long setting time and low early age strength [14,29]. Nevertheless, slag pastes activated with other common activator types such as alkaline hydroxides or silicates were reported to harden within a limited time (e.g., 3 hours) and reach high strength values (e.g., > 40 MPa). In the activation process of NC-activated slag systems, dissolved Ca^{2+} ions first react with CO_3^{2-} ions from the NC to form carbonate salts such as gaylussite and calcite. Over time, consumption of CO_3^{2-} ions increases the pH of the medium, followed by the dissolution of silicate species and the formation of a strength-giving phase of C-(A)-S-H [14]. It can be figured out that the reason for the delay in these reactions is the initial low alkalinity. Many studies attempted to increase the early age strength of NC-activated systems. Abdalqader et al. [12] studied the effect of MgO usage on NC-activated systems, and it was concluded that MgO incorporation accelerated the reaction rate. Ke et al. [9] reported that calcined layered double hydroxides can be used as carbonate binding agents. Incorporating calcined layered double hydroxides up to 10% (by wt.) significantly accelerates the kinetics of the reaction in sodium carbonate-activated slag pastes. Akturk et al. [16] found that low amounts of calcium hydroxide can enhance the early age and ultimate compressive strength of NCAS.

The use of reactive MgO as an alternative activator for slag was studied by many researchers [9, 30, 31]. The formation of a strength-giving phase of hydrotalcite with the incorporation of reactive MgO explains high strength in NC-activated slag systems. Bernal et al. [32] have a study on slag/metakaolin blends showing the effect of inclusion of MK in the binder. At high activator concentrations, the inclusion of a small amount of MK enhanced the compressive strength at early ages. In this study, a combination of reactive MgO and metakaolin presented the highest compressive strength (Fig. 3). The reason for this highest value might be about the formation of C-S-H gel and Al

or Na-enriched phase due to the metakaolin resource and formation of hydrotalcite and magnesium silicate hydrate (M-S-H) due to the reactive MgO resource. Although it is known that a denser internal structure can cause higher compressive strength since the M-S-H phases were believed to have limited contribution to strength [33], the effect of this dense structure on strength remains lower.

Chloride Migration Coefficient

NCAS mortars cured for 28 and 90 days under the ambient conditions were tested for non-steady state chloride migration, and the results are given in Figure 4 and Table 3.

The chloride penetration depths after silver nitrate sprayed were marked and the results for 90h days are presented on Figure 5. It can be easily seen that S mixes presented similar penetration depths through the sample and exhibited very low values. On the other hand, mixes containing M or MK showed inhomogeneous and higher values compared to S mixes. It can be concluded that since penetration depths were similar on S mixes, pore size distribution within this sample was homogeneous, while for other mixes were not.

Replacing slag with reactive MgO or a low amount of MK incorporation resulted in relatively lower chloride migration coefficients (15M and 10M5MK mixes) compared to others. However, only metakaolin replacement (15MK mixes) did not cause any decrease in the chloride migration results, reached significantly high values such as $31 \times 10^{-12} \text{ m}^2/\text{s}$ at 28 days. It can be concluded that reactive MgO may cause denser microstructure and lower permeability; thus, NCAS mixes can be modified by using M to provide chloride migration resistance. On the other hand, MK replacement at low amounts (10M5MK mixes) enhanced the compressive strength (Fig. 3) but did not represent the same performance in terms of chloride migration. It can be attributed to the non-homogeneous distribution of pores and the higher permeability of those mixes.

Over time, from 28 days to 90 days, all chloride migration coefficient values decreased for all mixes. The highest decrease was obtained in 15 MK values, from 31.10×10^{-12} to $8.50 \times 10^{-12} \text{ m}^2/\text{s}$. Only 5M10MK mixes presented similar values for both measurement times (Table 3).

Investigation of chloride migration is important especially for reinforced concrete durability due to the higher corrosion risk for reinforcing steel. Therefore, investigating the chloride migration of AAS systems is necessary to compare their chloride migration coefficients with the traditional cement-based systems. Table 4 compares the non-steady-state chloride migration coefficient values obtained in this study and the two other studies from the literature. The first one focused on chloride migration of OPC-based systems replaced with slag with replacement levels of 50%, 70%, and 85% [34]. Another study investigated the effects of M, layered double hydroxide, and calcined layered double

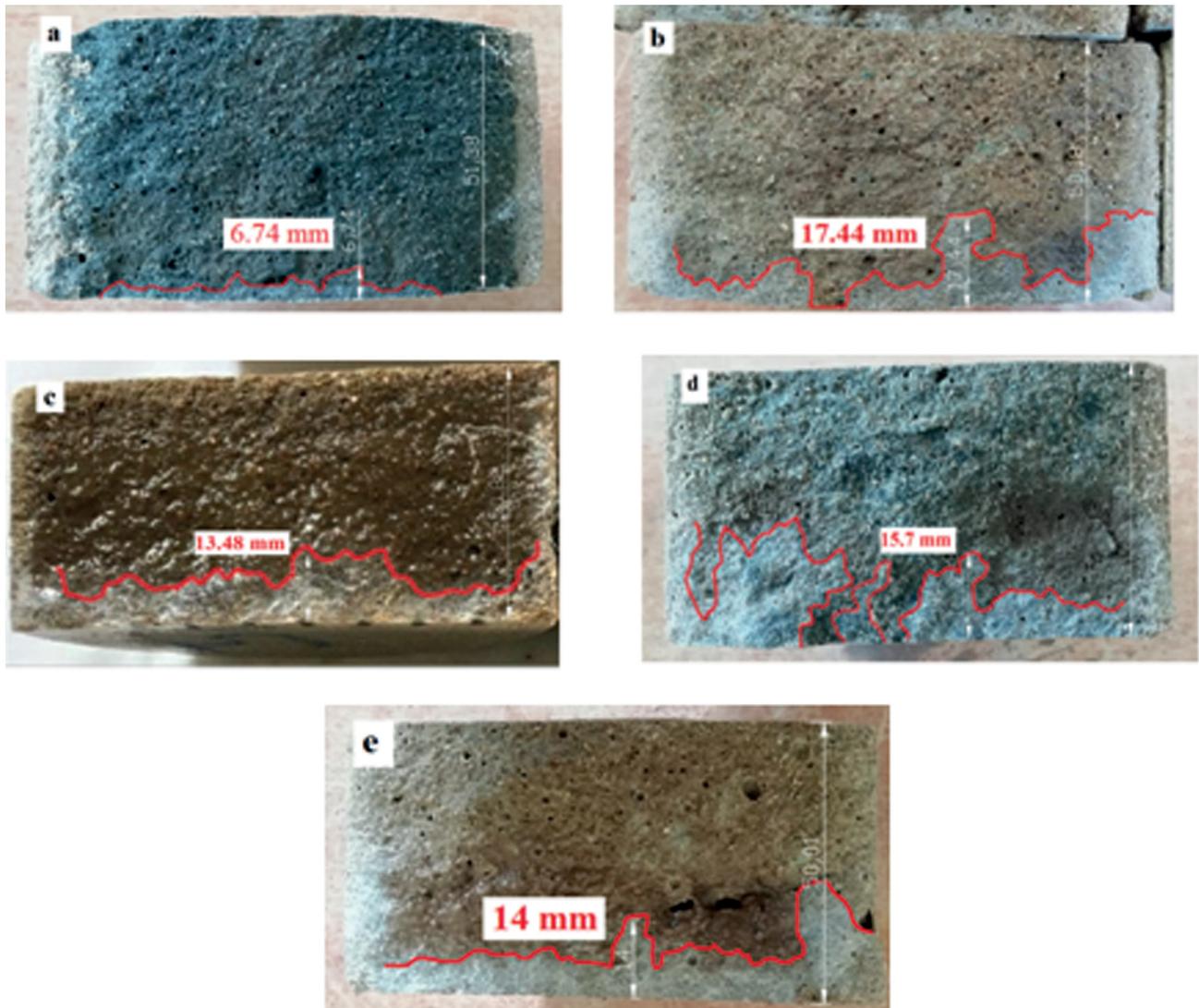


Figure 5. Chloride penetration depths after silver nitrate solution sprayed on 90th days samples (a) S, (b) 5M10MK, (c) 10M5MK (d) 15 M (e) 15MK.

Table 3. Average chloride migration coefficients ($D_{nssm} \times 10^{-12}$ (m²/s)) of mixes

Mixes	28 days	90 days
S	1.30	0.95
5M10MK	9.33	9.30
10M5MK	17.40	5.58
15M	5.89	4.11
15MK	31.10	8.50

hydroxide on the resistance of alkali-activated fly ash/slag (AAFS) mortars to chloride migration [24].

In each study, the resistance of AAMs was found as higher than that of OPC-based systems against chloride penetration. In the first study [34], it was found that chloride migration coefficients of mortars containing 50% and 70% slag decreased at 32% and 57%, compared to OPC-based mortars. In our study, samples containing a high level of M (15M mixes) reached lower values than that study. However, 5M10MK and 15MK mixes containing a high amount of MK did not reduce chloride migration and reached similar OPC values [34].

CONCLUSION

This study presents the compressive strength development and chloride migration of sodium

Table 4. Comparison of chloride migration coefficient results ($D_{nssm} \times 10^{-12} \text{ m}^2/\text{s}$) with other studies

Mortars	28 days	90 days	182 days	28 days		
	This Study		[34]		[24]	
S	1.30	0.95	BFS 50 (1)	7.92	M-A0	5.42
5M10MK	9.33	9.30	BFS 50 (2)	5.49	M-M2	4.68
10M5MK	17.40	5.58	BFS 70	4.30	M-M4	4.55
15M	5.89	4.11	BFS 85	5.38	M-OPC	7.55
15MK	31.10	8.50	OPC	9.67		

[34] BFS: blast furnace slag; numbers 50,70 and 85 indicate the replacement level of slag with OPC

[24] Mortars produced with M and the replacement ratios are 0, 2, 4%

carbonate-activated slag binder systems containing reactive MgO (M) and metakaolin (MK). The influences of the sole use and combination of these two additives on the compressive strength and chloride migration coefficient of sodium carbonate-activated slag binder systems were investigated.

Depending on the test results, concluding remarks are drawn as follows:

- The incorporation of a combination of M and MK at definite ratios was found to be an effective method to increase compressive strength significantly.
- The highest early and ultimate compressive strength values were obtained with a moderate amount of M (10%) and a low amount of MK (5%) replacement.
- More than 5% incorporation of MK caused a decrease in compressive strength and chloride penetration resistance.
- Different M and MK mix proportions may be used for further studies to reach optimum values in terms of high compressive strength and low chloride migration coefficient.
- To clarify the behavior of mixes for compressive strength and chloride migration performances, a detailed microstructural analysis should be carried out.

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AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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