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Comparing a natural pozzolan, zeolite, to metakaolin and silica fume in terms of their effect on the durability characteristics of concrete: A laboratory study

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HIGHLIGHTS

- ▶ Optimal replacement level is 7.5–10% for silica fume, 10–15% for metakaolin and approximately 10% for zeolite.
- ▶ Utilizing pozzolans is more effective in enhancing the durability of concrete than lowering w/c ratio.
- ▶ Because zeolite is environmentally friendly, it could be a good substitute for silica fume and metakaolin.

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ABSTRACT

The durability of concrete structures and, consequently, their service life, have improved significantly due to the introduction of pozzolans to the industry; thus, using newly developed pozzolans has become a necessity for the construction of concrete structures, especially in aggressive environments. In the laboratory study presented here, the effects of substituting cement with 10%, 20% and 30% natural zeolite on concrete durability were compared to the effects of substituting 5%, 10% and 15% metakaolin and 5%, 7.5% and 10% silica fume, along with water-to-cement ratios of 0.35, 0.40, 0.45 and 0.50. The comparison is made according to the test results of compressive strength, water absorption, sorptivity, volume of voids, electrical resistance, gas permeability and chloride diffusion, and their inter-relationships also were investigated. Results show that, in general, the zeolite is not as active as silica fume or metakaolin, although it could be used as a substitute for pozzolans because it has better durability characteristics and is economical and environmentally friendly as well.

1. Introduction

Using pozzolans has become one of the most common ways to increase the service life of concrete structures. Pozzolans primarily affect the pore structure refinement of concrete, which leads to higher strength and lower permeability [1,2].

Two of the most commonly used pozzolans in industrial projects are metakaoline and silica fume (SF). According to several studies, both of these pozzolans used at the same replacement level can sharply increase the compressive strength of concrete and decrease its permeability [3,4]. However, in recent years, the industry has shifted to using natural pozzolans because of their lower cost and accessibility. One of the most common natural pozzolanic materials is natural zeolites.

Zeolite is a crystalline, hydrated alumino-silicate of alkali and alkaline earth cations having infinite, open, three-dimensional structures. Natural zeolite has a cryptocrystalline structure, and like other pozzolans, it undergoes pozzolanic activity due to its high quantity of reactive SiO_2 and Al_2O_3, which combines with $Ca(OH)_2$ to form additional C–S–H gel [5]. The presence of zeolite can retard the hydration process, thereby reducing the permeability, sorptivity and diffusivity of concrete because it reduces porosity and improves the transition zone structure between the blended cement paste and the aggregate [6–9]. Some of the main properties of natural zeolites include ion exchange, their large surface area and their ability to act as molecular sieves because their pore structures have molecular dimensions of variable sizes that can hold some molecules or ions, but not others. For example, zeolites are used extensively for ion exchange in water purification and softening, as well as in chemistry as drying agents. Many researches investigating these characteristic shave tested the use of zeolites in the concrete industry as a replacement for cement. Using thermo-gravimetric and XRD analysis, these studies have shown that the pozzolanic reaction in pastes containing zeolite (clinoptilolite type) considerably reduced the calcium hydroxide content [10]. In addition, the comparison of cement mortar containing clinoptilolite to reference cement mortar confirmed that

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the water demand, soundness and setting times of the cement pastes increased as the clinoptilolite content increased, while clinoptilolite replacement reduced the water absorption and porosity of the mortars [11]. Also, a number of studies have compared zeolite to other pozzolanic materials, indicating that the activity of natural zeolite is superior to fly ash but poorer than SF [12]. Test results by Ahmadi [13] revealed that the pozzolanic activity of natural zeolite is lower than silica fume but that there is a high rate of lime consumption. Also, the increase of compressive strength in concrete containing natural zeolite was not higher than in SF, although this parameter for materials is higher than for the reference concrete. Other studies have been conducted on the effects of natural zeolite on concrete durability. Findings from these studies indicate that using natural zeolite diminishes the formation of alkali–silica reaction (ASR) gel, though not as well as ground granulated blast-furnace slag (GGBFS), and protects the composite material against sulfate attacks by diminishing the formation of ettringite [14]. Moreover, a comparison between the effects of natural zeolite, slag and SF on the prevention of the ASR indicated that natural zeolite operates as effectively as fly ash and better than slag, but not as well as SF [15]. Other researchers also have confirmed the decreasing effectiveness of natural zeolite on ASR [10,13,16]. Investigations on chloride diffusivity and initial surface absorption have shown that natural zeolite is better than fly ash but not as good as SF [17]. Pargar et al. [18] reported that using zeolite in splash and tidal zones decreases chloride diffusion. Their results showed that adding 20% and 30% zeolite as a replacement for cement reduces chloride penetration up to 61% in splash zones and up to 81% in tidal zones in comparison with control concrete, but it demonstrated less efficiency than SF. In addition, an gas permeability experiment revealed that a 10% zeolite replacement reduced gas permeability by approximately 25% [16]. Additionally, studies on the ternary combination of natural zeolite with SF and fly ash on the transport properties of concrete indicated that this combination is better than fly ash or SF alone [19].

In this paper, the behavior of concrete is discussed in terms of factors such as compressive strength, electrical resistivity, water absorption, sorptivity, gas permeability, volume of voids and chloride diffusivity. A laboratory study was conducted to compare the performance of concrete containing zeolite (10%, 20% and 30%) with different w/c ratios (0.35, 0.40, 0.45, 0.50) to 5%, 7.5% and 10% SF and 5%, 10% and 15% metakaoline in terms of their durability functions.

2. Experimental program

2.1. Materials

The materials used throughout the experiment include type II Portland cement combined with zeolite, SF and metakaolin. The fine and coarse aggregates used for casting the concrete mixture were river sand and crushed limestone at ratios of 38% and 68%, respectively. The coarse aggregate has a maximum size of 19 mm. Table 1 shows the composition of these materials.

Table 1
Chemical properties of binders.

Oxide comp. (% by mass)	PC	Gravel	Sand	Natural zeolite	Silica fume	Metakaolin
SiO_2	21	7.88	50.73	67.79	93.16	51.58
Al_2O_3	5	0.53	5.91	13.66	1.13	43.87
Fe_2O_3	3.5	1.07	7.45	1.44	0.72	0.99
CaO	63	44.12	15.16	1.68	–	0.2
MgO	1.8	4.61	7.33	1.2	1.6	0.18
SO_3	1.6	0.1	0.003	0.5	0.05	–
Na_2O	0.5	0.03	1.46	2.04	–	0.01
K_2O	0.6	0.36	0.65	1.42	–	0.12
LOI	2	40.86	10.21	10.23	1.58	0.57

Table 2
Details of the concrete mixtures.

Sample	w/b	Cement (kg/m^3)	Supplementary cementitious materials (kg/m^3)	Water (kg/m^3)	Super plasticizer (kg/m^3)	Slump (cm)
C1	0.35	400	–	140	0.35	8
C2	0.40	400	–	160	0.2	7
C3	0.45	400	–	180	0.1	15
C4	0.50	400	–	200	0	18
ZE1	0.40	360	40	160	4	6
ZE2	0.40	320	80	160	8	8
ZE3	0.40	280	120	160	14	6
MK1	0.40	380	20	160	1.2	6
MK2	0.40	370	30	160	1.4	5
MK3	0.40	360	40	160	1.6	8
SF1	0.40	380	20	160	0.8	5
SF2	0.40	360	40	160	1.4	5.5
SF3	0.40	340	60	160	1.6	8

2.2. Mixture proportions

To examine the effects of the w/c ratio on concrete properties, four specimens were prepared with different w/c ratios of 0.35, 0.40, 0.45 and 0.50, without the addition of any mineral admixtures. Also, concrete mixtures incorporating different replacement levels of natural zeolite, SF, and metakaolin were made, with a constant cementitious materials content of 400 kg/m^3. The mixing process was conducted according to ASTM C93, and afterwards, a cement blend of mixing water, super plasticizer and pozzolanic materials with powder was added to the mixture. Poly-carboxilate based super plasticizer was used to achieve the desired workability.

All compressive strength specimens were moist cured for 28 days; after demolding, the other specimens were moist cured for 2 days and then were placed at room temperature for up to 28 days under laboratory conditions before the specified experiments were executed. Table 2 shows the proportion of materials mixed to form the concrete specimens.

Natural zeolite has a three-dimensional frame structure that contains many pores with a high surface area [16]. Therefore, a super plasticizer must be added to concretes containing natural zeolite in order to maintain the slump of concrete, as interpreted in Table 2. Specimens containing SF and metakaolin do not require the same amount of additional super plasticizer to maintain the appropriate slump.

3. Test procedure

The concrete specimens were tested for compressive strength, electrical resistivity, water absorption, sorptivity, gas permeability, chloride diffusivity and void content.

3.1. Compressive strength

Compressive strength test was conducted on the cube specimens 150×150×150 mm in size at 7 and 28 days of moist curing.

Fig. 1. Gas permeability apparatus.

3.2. Electrical resistivity

The electrical resistivity of concrete was measured on 10 cm cubes in compliance with the AC Impedance Spectrometry (ACIS) method using a concrete resistance meter [20,21]. A frequency of 1.1 kHz was applied on the saturated surface of the dried specimens.

3.3. Gas permeability

Three cylindrical specimens 150 mm in diameter and 300 mm in height were obtained from each concrete mixture after the concretes were cured for 28 days. The test was determined using regime A of the Cembureau method (Fig. 1) [22]. The specific permeability coefficient of the specimens was calculated according to the Hagen–Poiseuille relationship [23].

3.4. Sorptivity

Sorption occurs when concrete comes into contact with water or air moisture. After 180 days, the water sorptivity of the 100 mm cubed concrete specimens was determined according to RILEM-CPC-11.2 [24].

3.5. Water absorption and volume of voids

Water absorption tests were conducted for each concrete mixture at 28 days on 300 mm cubes according to both BS1881-122 [25] and ASTM C642 [26] to allow a comparison between the two. The volume of voids and water absorption were determined based on the steps described in ASTM C 642.

3.6. Chloride diffusion

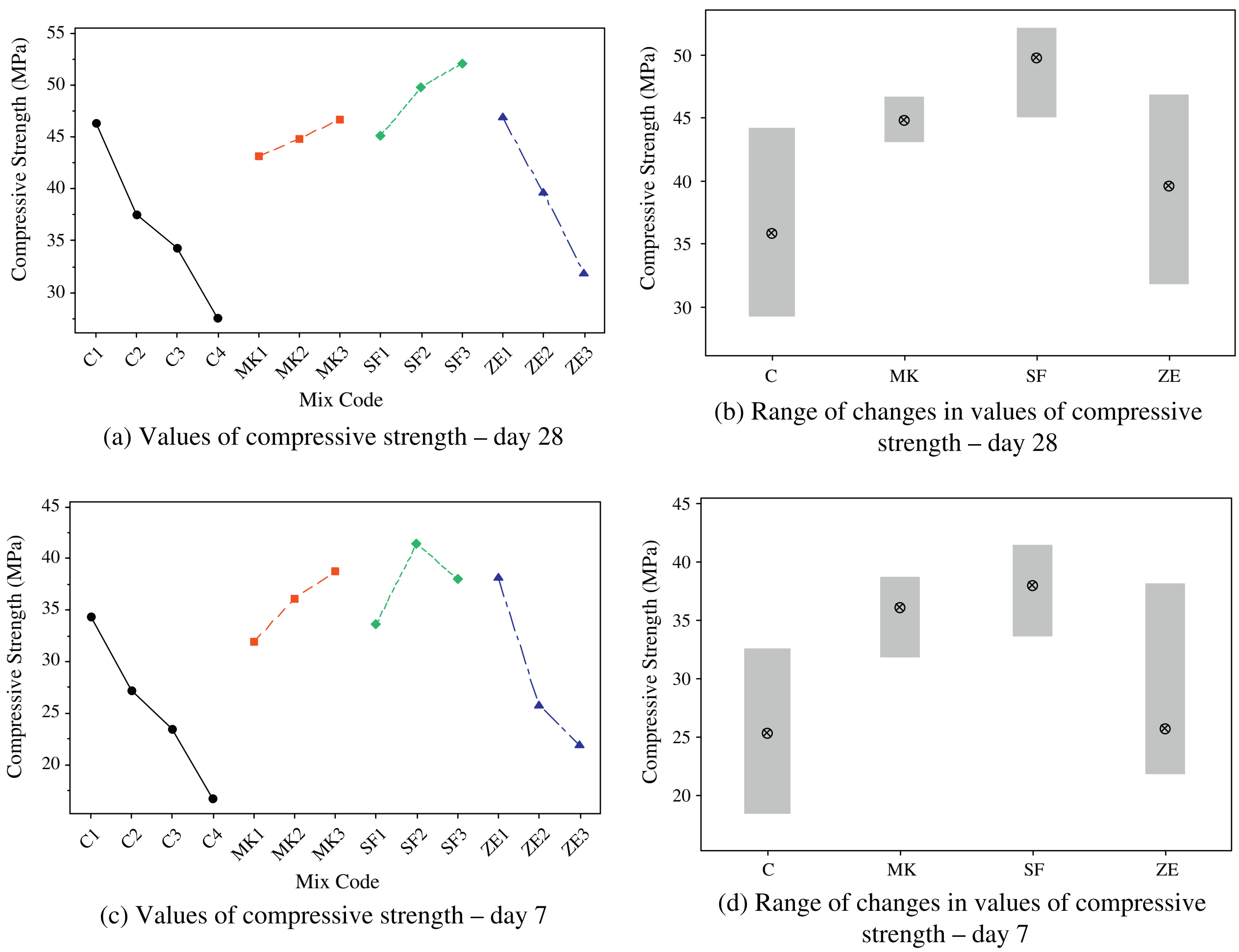
For each mixture, a cylindrical sample 150 mm in diameter and 300 mm in height was prepared and, after curing, was cut into three cylinders with a thickness of 100 mm. Then, the chloride diffusion test was conducted in compliance with NT Build 443 [27]. This Nordtest method specifies a procedure by which to determine the penetration parameters for estimating the resistance of these cylinders to chloride penetration into hardened concrete or other cement-based materials. The acid-soluble chloride content of each sample was analyzed according to ASTM C 114 [28]. The chloride ion diffusion coefficient, D_a, and surface concentration, C_s, were calculated using Fick's second law [29,30].

4. Result and discussion

The test results are presented in the following subsections.

4.1. Compressive strength

Fig. 2a, c illustrates the compressive strength of all specimens at 28 and 7 days under standard conditions. A comparison of the concrete containing pozzolans with the control concrete showed an increase in the compressive strength of the former at 7 and 28 days, except for in the ZE2 and ZE3 specimens at 7 days and the ZE3 specimen at 28 days. The compressive strength varies widely in



(a) Values of compressive strength – day 28

(b) Range of changes in values of compressive strength – day 28

(c) Values of compressive strength – day 7

(d) Range of changes in values of compressive strength – day 7

Fig. 2. Results of the compressive strength test.

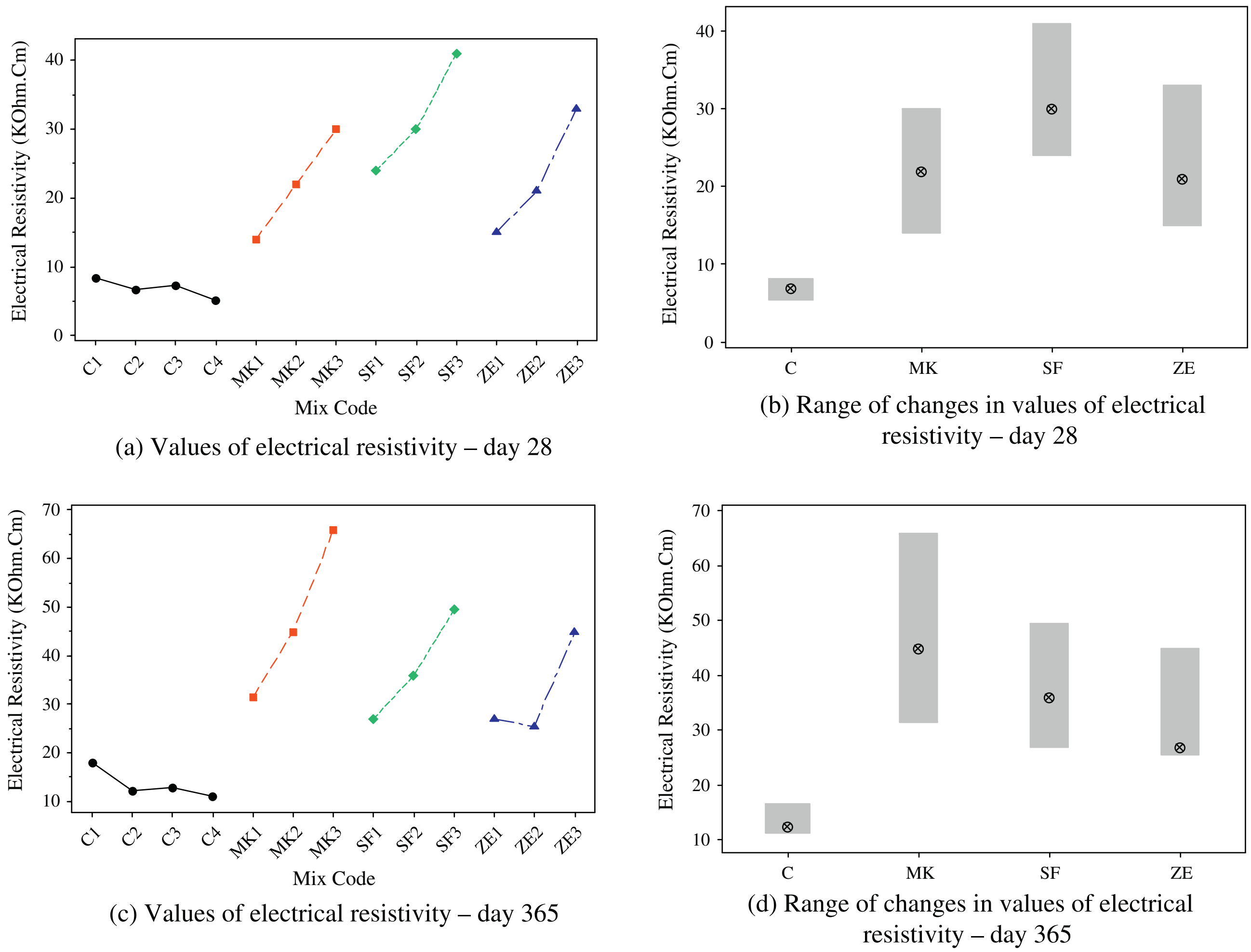


Fig. 3. Results of the electrical resistivity test.

concretes with zeolite but is lower than that of concrete with different water-to-cement ratios and equivalent to that of concrete containing metakaolin at its highest value. This could be due to the weak and brittle structure of zeolite at 20% and 30% and its strong structure at 10%. Ahmadi and Shekarchi [16] reported that the optimum amount of zeolite replacement for 90 days of compressive strength is 15%. However, the addition of SF leads to higher compressive strengths in concretes.

4.2. Electrical resistivity

The electrical resistivity was measured at 28 days and 365 days, and the results are demonstrated in Fig. 3a, c. The specimens had been placed in water for 48 h before testing and thus were nearly saturated when the electrical resistivity was measured. The electrical resistivity increases with the further addition of pozzolans, which implies that the sensitivity of electrical resistance depends

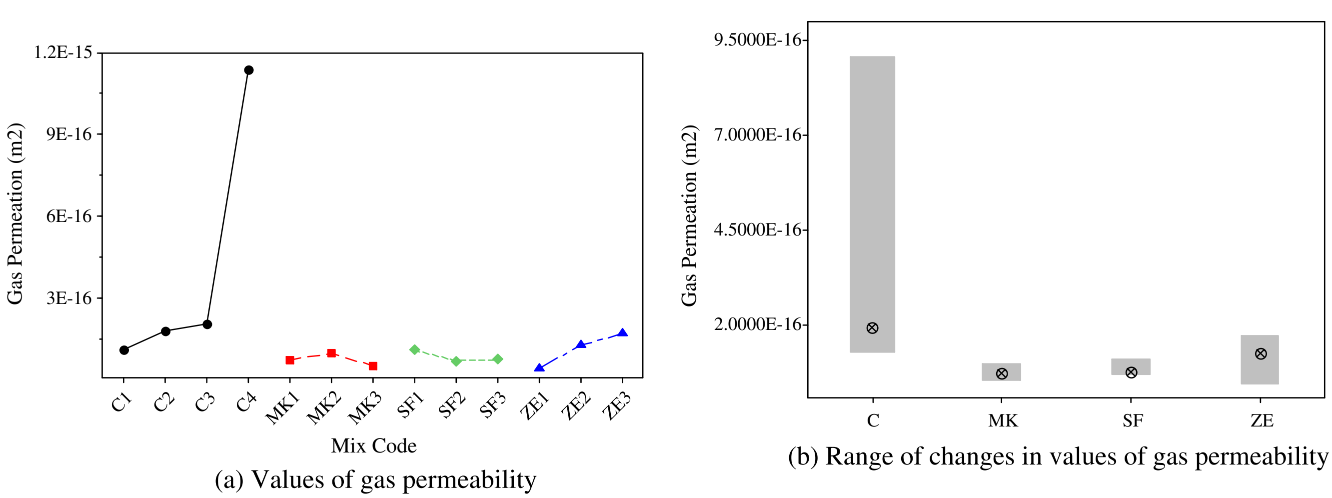


Fig. 4. Results of the gas permeability test.

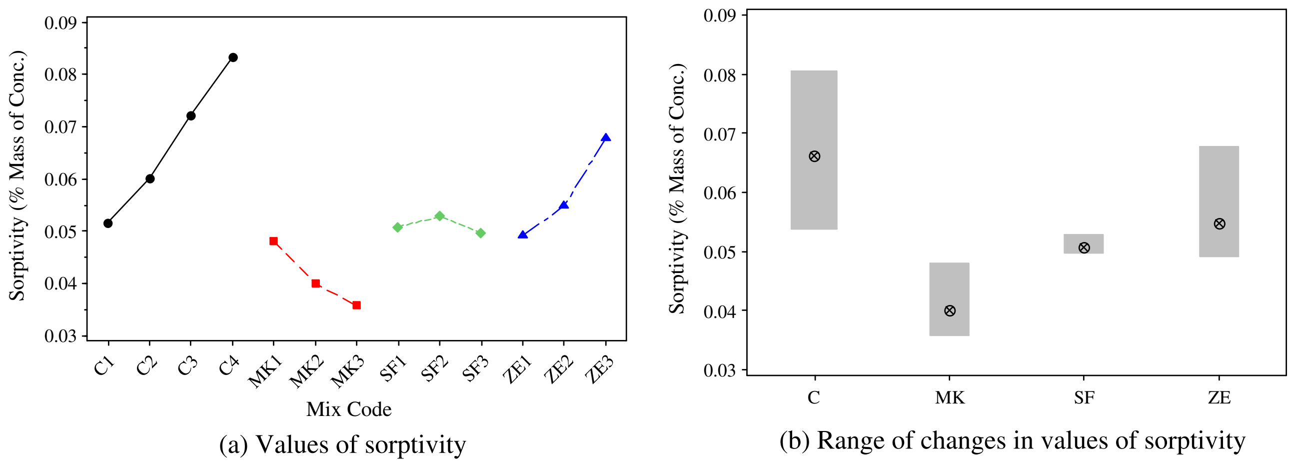


Fig. 5. Results of the sorptivity test.

on the amount of pozzolan used. Zeolite shows resistivity lower than that of SF and equal to that of metakaolin, while SF has the highest electrical resistivity. This could be due to zeolite's complex structure. Despite being natural pozzolan, its performance is acceptable in terms of electrical resistivity. The control specimen shows low resistivity, and no significant difference was observed upon altering the water-to-cement ratio.

4.3. Gas permeability

The gas permeability of the concrete specimens is illustrated in Fig. 4. The gas permeability of all concrete specimens containing pozzolans decreased in comparison with that of the control concrete.

For specimens containing zeolite, it is clear that higher permeability is achieved when the zeolite content increases, as reported by Ahmadi and Shekarchi [16]. This could be due to the fact that zeolite is a natural pozzolan with slow reaction activity and thus has a short curing time and a higher replacement level; this causes incomplete secondary reactions, leading to the conglomeration of zeolite. Among specimens containing metakaolin, MK2 reached the highest value, whereas MK3 showed the lowest amount of oxygen transfer. According to Fig. 4a, oxygen permeated more through the SF1 specimen, while the SF2 and SF3 specimens showed the same amount of gas permeability. As expected, no uniform trend appeared among concretes containing SF and metakaolin in terms of gas permeability.

4.4. Sorptivity

Fig. 5a, b shows the specimens' sorptivity results, indicating that the performance of zeolite varies as increasing replacement levels raise the sorptivity of the specimens. No significant difference exists between specimens with SF and metakaolin. Sorptivity decreases as their placement percentage increases. As Fig. 5 illustrates, increasing the w/c ratio increases the capillary absorption coefficient.

4.5. Water absorption and volume of voids

Fig. 6a, c, and e illustrates the water absorption based on ASTM, BS (30 min) and BS (24 h) methods respectively and Fig. 6g shows the void volume results. The same trend is obtained in both methods used to determine water absorption.

The half-hour water absorption of concretes varies significantly in specimens with different water-to-cement ratios. The rate of permeation in the specimen with a water-to-cement ratio of 0.35 is lower than that of SF3. Zeolite has higher water permeation than SF or metakaolin due to its permeability factor.

The lower bound of 24 h of water absorption in different water-to-cement ratios is almost equal to the upper bound of specimens with SF. The 24 h of water absorption is highly dependent on the amount of capillary pores and plays a more important role in water permeation, causing the specimens containing SF and metakaolin to permeate less water. On the other hand, the 24 h of water permeation has a wider range, which shows the effect of the amount of pozzolan used in concrete.

Specimens containing SF with different levels of substitution show less variation in half-hour permeations than the other two pozzolans. This indicates that SF is influenced more by the curing condition than the other two pozzolans, leading to micro cracks on the surface of the concrete and therefore increasing water permeation in specimens with SF and other pozzolans in comparison with a water-to-cement ratio of 0.35. Further use of zeolite has a greater influence on half-hour permeations due to its higher permeability factor.

The porosity of concretes containing zeolite is higher than that of the other two pozzolans. Also, the porosity varies more with water-to-cement ratio alterations. In other words, the water-to-cement ratio greatly influences the porosity. Concretes containing SF and metakaolin have the lowest porosity because they have fewer capillary pores.

The lowest amount of porosity in concrete containing zeolite is equivalent to the highest amount in concretes containing SF and metakaolin, while the lowest amount of gas permeation in zeolite (ZE1) is even lower than that of SF and metakaolin. This indicates the very low permeability of ZE1 in spite of its higher porosity. Increasing the replacement level increases the gas permeability factor. This can be attributed to the complex microstructure of zeolite in this mixture's design.

Fig. 6 shows that the use of zeolite at 20% slightly increased the water absorption and porosity, which is undesirable, but increasing this percentage even more caused a slight reduction in water absorption. On the other hand, increasing the replacement percentages of SF and metakaolin causes a small decline in water absorption and porosity.

4.6. Chloride diffusion

Chloride diffusion coefficient and surface chloride for each specimen at 9 months are presented in Fig. 7a, c, respectively. The amount of *D* variation in concretes containing zeolite is lower than in the other concretes, and its lowest amount is higher than the other two pozzolans. However, after 9 months of exposing the specimens to chloride penetration, their diffusion factor decreases with an increase in the replacement levels of metakaolin and zeolite, while the amount of surface chloride increases with an increase in there placement percentage of the pozzolans. The diffusivity factor of specimens containing SF decreases with an increase in the percentage from 5 to 7.5 and from 7.5 to 10. The diffusion coefficient in concretes with different water-to-cement ratios is at least two times higher than in those containing pozzolans. Also, the upper boundary of surface chlorides in concretes with different water-to-cement ratios is almost equivalent to the

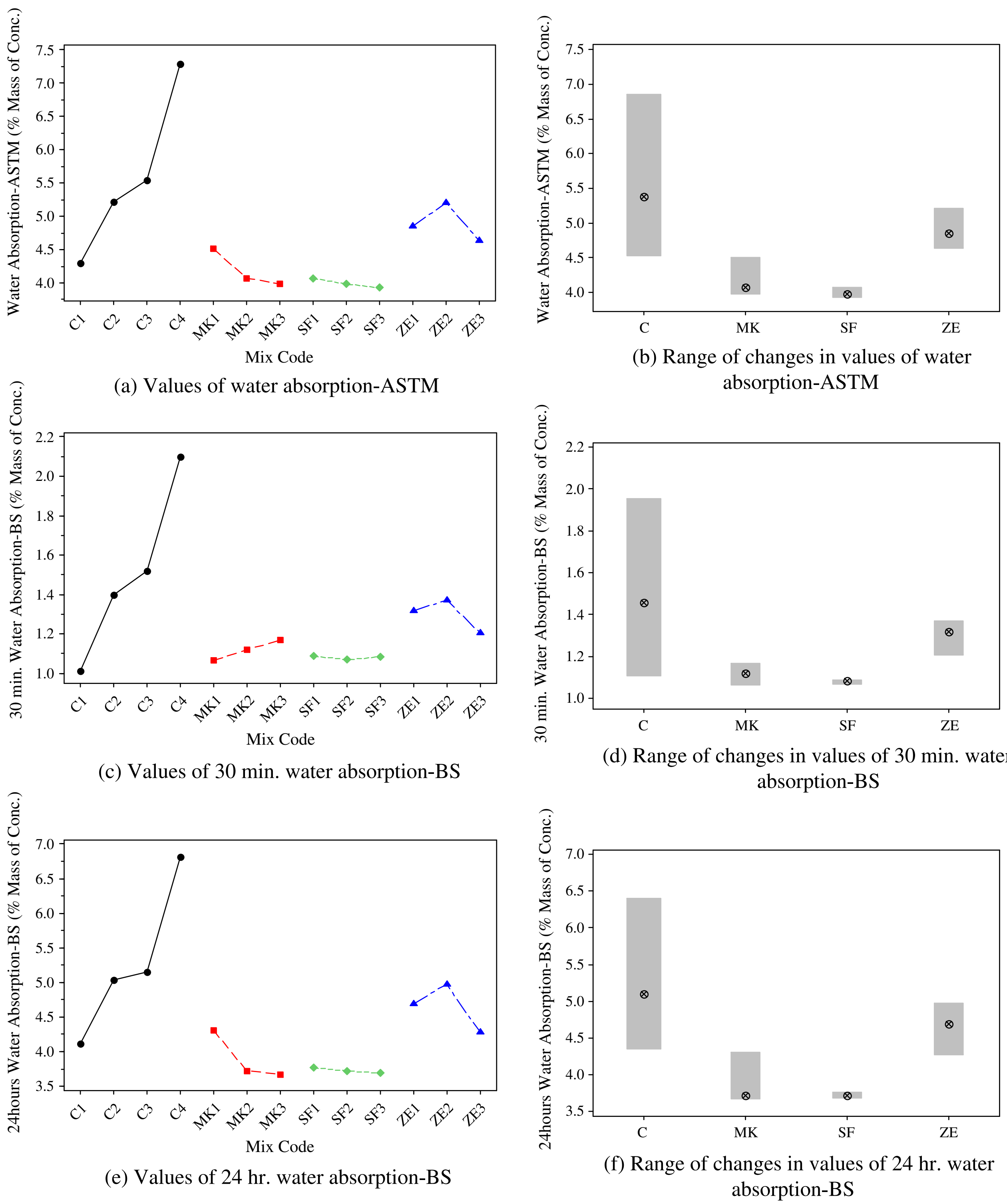
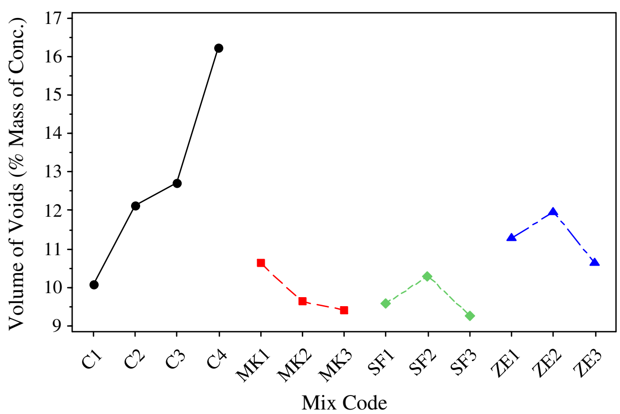
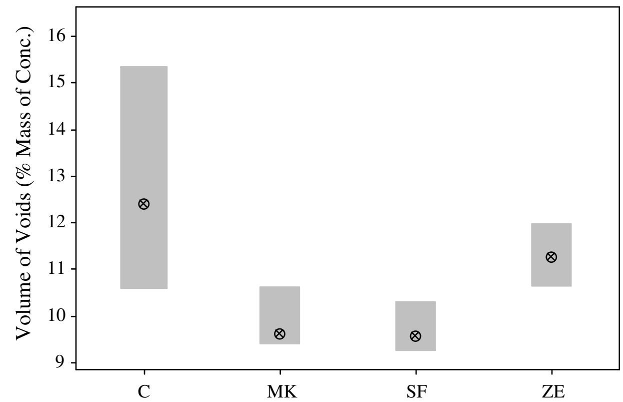


Fig. 6. Results of the water absorption and volume of voids tests.



(g) Values of volume of voids



(h) Range of changes in values of volume of voids

Fig. 6. (*continued*)

lower boundaries in concrete containing pozzolans. However, the amount of surface chloride does not show any significant change with increases in the water-to-cement ratio, while it significantly increases with increases in metakaolin. Also, increasing zeolite replacement from 10% to 20% sharply increases the amount of surface chloride from 20% to 30%. For SF, surface chloride increases when the SF replacement level increases from 5% to 7.5% and decreases when the SF replacement level increases from 7.5% to 10%. This can be attributed to the effect of the curing condition on SF3; due to the higher percentage of SF, the amount of cracks on the surface of the concrete increases, causing the value of *D* to increase and the value of *C* to decrease in comparison with SF2.

The results obtained from the chloride content of specimens at a depth of 20 mm, presented in Fig. 7e were selected as the basis of comparison in order to obtain more consistent conclusions. For all concretes containing pozzolans, the amount of chloride at a depth of 20 mm was lower than that of recorded in the control mixture.

In zeolite concretes, the higher the replacement level, the less chloride at a depth of 20 mm. Thus, specimen ZE1 displayed the highest amount of chloride at 20 mm. Among the silica fume mixes, SF2 had the lowest amount of chloride at a 20 mm depth. None of the mixtures with metakaolin showed a considerable difference in the amount of chloride at 20 mm, although specimen MK3 had the lowest chloride content. A comparison of different w/c ratios shows that the amount of chloride at a depth of 20 mm increases with increases in the w/c ratio; therefore, C1 has the lowest chloride content among specimens with different water-to-cement ratios but the highest chloride content in comparison with specimens containing pozzolans.

4.7. Correlation between durability parameters and compressive strength

The correlation between the durability properties of different mixtures containing different percentages of pozzolans and the compressive strength was determined using linear and exponential regressions. The results are summarized in Table 3. In terms of gas permeability, silica fume correlates well with values of R^2 higher than 90%. The results of water absorption show a very low coefficient of determination ($R^2 < 62\%$) for zeolite and metakaolin. In general, the values for concretes containing zeolite correlate poorly with durability properties, which indicate that zeolite does not follow the trend.

4.8. The influence of replacement level on concrete properties

Table 4 reports the effect of increasing the replacement levels of different pozzolans on concrete properties. Further use of zeolite effectively increases electrical resistivity and decreases chloride diffusivity, but it is less effective in decreasing water absorption, which seems to be the case with the other two pozzolans as well. A higher replacement level of silica fume leads to higher compressive strength and electrical resistivity and lower gas permeability and chloride diffusivity, while no significant effect was observed on sorptivity reduction. Using metakaolin is effective in terms of all concrete properties except for water absorption, as mentioned previously.

4.9. The influence of same replacement level of pozzolans

Fig. 8 shows the influence of different pozzolans at the same replacement level on different properties of concrete. Results obtained from the 10% replacement level show that zeolite performs better for water absorption (BS and ASTM) and chloride diffusivity, SF performs better for compressive strength and electrical resistivity, and metakaolin performs better for gas permeability. The performance of SF and zeolite regarding sorptivity is quite the same. Also at this replacement percentage, all three pozzolans behave similarly in terms of the volume of voids. Therefore, zeolite would be a good substitute for SF and metakaolin at 10% replacement because it is natural and less expensive.

4.10. Durability of concretes with the same compressive strength

Specimens ZE1, SF1, MK1 and C1 have the same compressive strength, 45±2 MPa. Fig. 9 shows the influence of the compressive strength of the various pozzolans on different properties of concrete. The results of performance testing of these pozzolans shows that ZE1 and MK1 positively affect sorptivity, ZE1 positively affects gas permeability, SF1 positively affects electrical resistivity, C1 positively affects water absorption-Bs and void volume, and SF1 positively affects water absorption-ASTM. It can be concluded that for concretes with the same compressive strength, the replacement of pozzolans is more effective than lowering the w/c ratio in enhancing concrete durability.

It also is clear that using pozzolans improves electrical resistivity, chloride diffusivity, sorptivity and, to a lesser extent, gas permeability. Zeolite has obvious benefits in terms of chloride

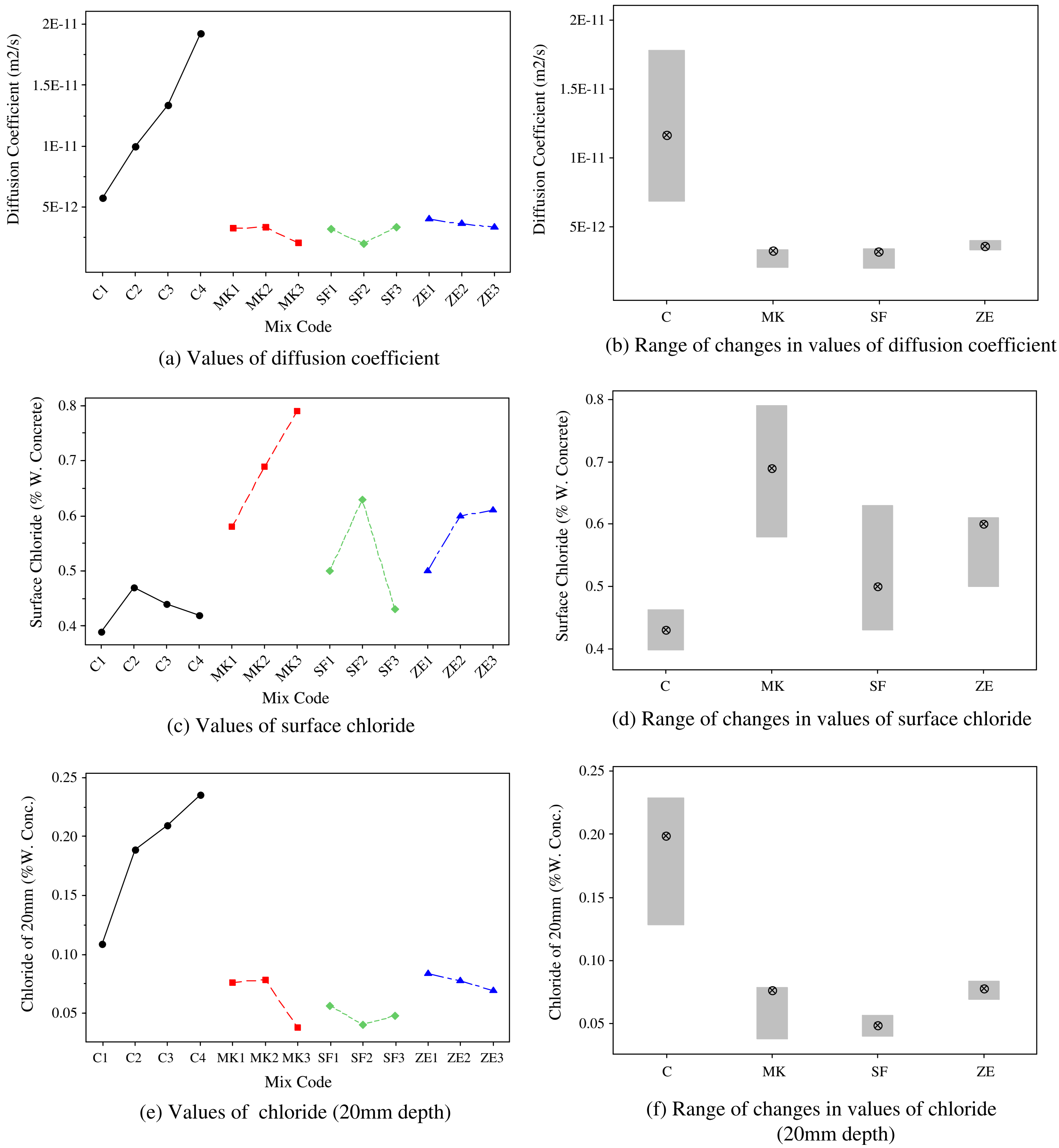


Fig. 7. Results of the diffusion coefficient, surface chloride and chloride at depth of 20 mm.

diffusivity, gas permeability and sorptivity reduction. While these are important parameters in the corrosion process, using zeolite does not change the water absorption or void volume values. Low sorptivity indicates that the capillary absorption is very low, while high water absorption and void volume indicate the existence of many pores. Therefore, it can be concluded that using zeolite reduces the amount of capillary pores without changing the total volume of pores considerably.

5. Conclusion

In this study, the effects of substituting cement with 10%, 20% and 30% natural zeolite were compared to the effects of substituting 5%, 10% and 15% metakaolin and 5%, 7.5%, 35% and 10% silica fume, along with water-to-cement ratios of 0.35, 0.40, 0.45 and 0.50 in terms of their durability functions. The main conclusions that can be drawn from this study are as follows:

Table 3
Regression formulations for transport properties (y) vs. compressive strength (x).

Equation	G.P.			W.A.			E.R. (28-day)			*D*		
	ZE	MK	SF	ZE	MK	SF	ZE	MK	SF	ZE	MK	SF
Linear: $y = a.x + b$												
a	−0.09	−0.13	−0.08	0.01	−0.03	−0.02	−0.97	2.40	2.19	−0.04	−0.9	−0.55
b	4.83	6.82	4.73	1.08	2.43	2.18	56.74	−85.18	−75.66	6.12	45.1	29.62
R^2	0.80	0.88	0.96	0.21	0.61	0.81	0.30	0.89	0.97	0.00	1.0	0.82
Exponential: $y = a.e^{bx}$												
a	42.87	156.52	21.09	1.09	3.15	2.65	67.43	0.01	0.08	3.57	17588.0	548.91
b	−0.09	−0.12	−0.07	0.01	−0.02	−0.02	−0.04	0.16	0.12	0.00	−0.2	−0.11
R^2	0.80	0.84	0.96	0.22	0.58	0.81	0.11	0.98	0.96	0.00	1.0	0.84

Table 4
The Influence of Replacement level on concrete properties.

Property	Influence of increasing the replacement level of zeolite	Influence of increasing the replacement level of silica fume	Influence of increasing the replacement level of metakaolin
Compressive strength	↓	↑	↑
Electrical resistivity	↑	↑	↑
Water absorption (BS)	Constant	Constant	Constant
Water absorption (ASTM)	Unknown	Constant	↑
Void volume	Unknown	Constant	↑
Sorption	↓	Constant	↑
Gas permeability	↓	↑	↑
Chloride diffusion	↑	↑	↑

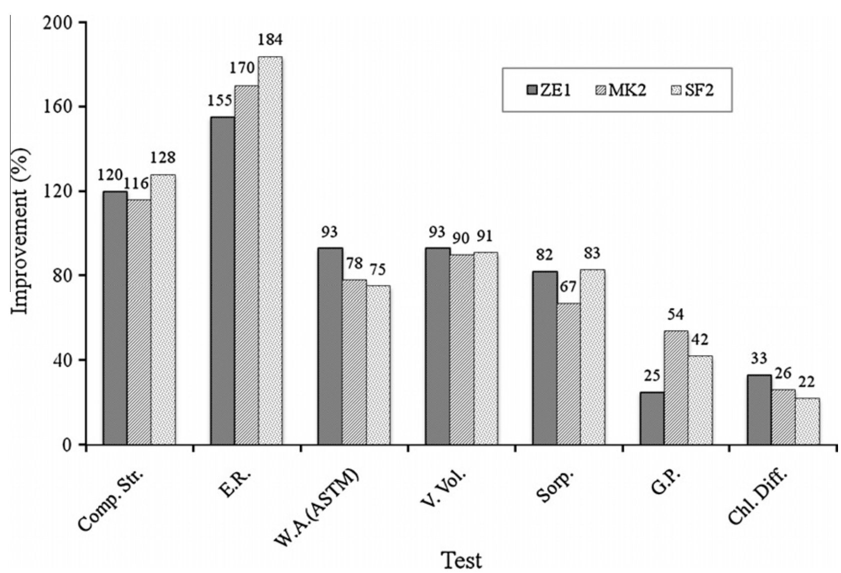


Fig. 8. The influence of same replacement level on concrete properties.

1. The results show that the optimal replacement level is 7.5–10% for silica fume, 10–15% for metakaolin and approximately 10% for zeolite.
2. Although zeolite is a natural pozzolan that requires long-term curing to complete the hydration process, after 3 days of moist curing, this pozzolan at a 10% replacement level produced acceptable performance in terms of concrete durability.
3. Because zeolite is more cost effective, accessible, and natural (environmentally friendly), it seems that it could be a good substitute for silica fume and metakaolin.
4. The surface chloride displayed an inverse relationship with the amount of chloride at a depth of 20 mm in concrete.
5. The inter-relationship between the durability parameters implies a proper correlation due to their fine microstructures. The weak correlation for zeolite can be attributed to its tortuous micro structure.
6. The use of pozzolans at an effective replacement level was found to be more efficient than lowering the w/c ratio in enhancing the durability of concrete.

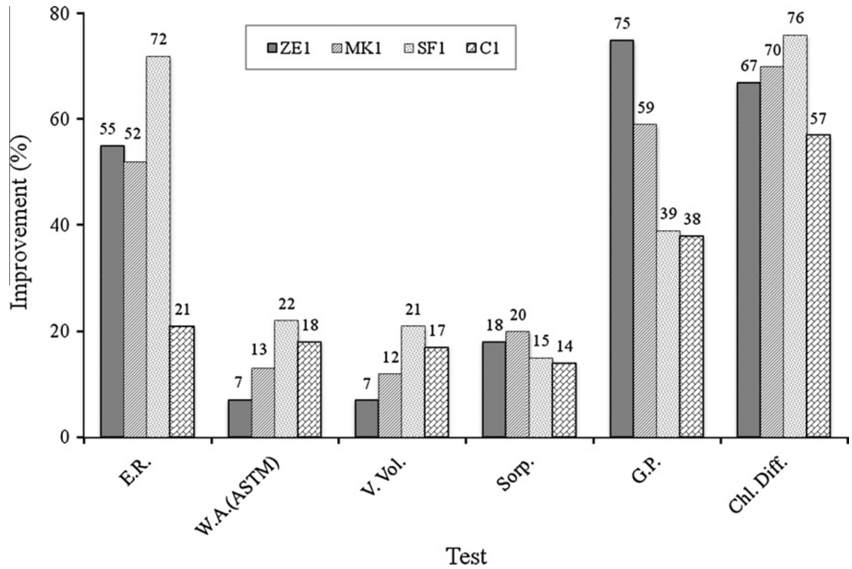


Fig. 9. The influence of pozzolans with same compressive strength on concrete properties.

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