



Research Article

Chemical resistance of hardened mortar containing andesite and marble industry waste powder

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ABSTRACT

The sludge generated during forming processes of marble and andesite rocks is kept in dust form after drying. Due to the high consumption of andesite and marble, the storage and health problems of these dusts arise. Therefore, reducing the environmental impacts of waste and recovering them for the economy is an important issue. For this purpose, in this work, mortar specimens were manufactured using 0%, 5%, 10%, 15% and 20% of waste marble and andesite powders separately by Portland cement. Strength properties of the samples were investigated before and after immersion to the hydrochloric acid (HCl), sodium sulfate (Na₂SO₄) and magnesium sulfate (MgSO₄) solutions. The results indicated that partial substitution of Portland cement by andesite and marble powder up to 10% have positive influence on the mechanical properties of the mortars at ambient conditions. In addition, the andesite incorporated mortars have the better performance under the acid and sulfate environments than the other mortars. On the other hand, substitution of Portland cement by marble powder more than 5% has negative influence on the chemical resistance of the mortars.

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1. Introduction

Greenhouse gases along with the natural resources issue are important role on the sustainable development of construction industry. Construction industry is researching for the utilization of substitute alternatives for cement manufacture. In the last decades, there are studies focused on substitution of cement with several wastes such as fly ash, blast furnace slag, rice husk ash, and etc. (Rana et al. 2015; Naik 2008). The CO₂ emission originated from the cement manufacture may also be limited by restricting it with such wastes (Yang et al. 2015; Rana et al. 2015).

Approximately, 7 million tons of natural rock is processed in Turkey for each year, 75% of which is formed at five thousand quarries. Andesite is a type and subtype volcanic and magmatic rock, respectively. Andesite is employed for several architectural and civil engineering applications in Turkey as well as in other countries (Sarisik et al. 2011; Davraz et al. 2018). Powder and sand are

disposed as by products during polishing and cutting processes of rocks for several aims. A large-scale andesite manufacture generates vast amount of waste powder; nearly one quarter of the andesite and marble is discharged during polishing, cutting, mining and other applications. Discharge of andesite and marble processing waste powder into the environment can lead to significant health and environmental problems (Soğancıoğlu et al. 2013; Davraz et al. 2018; Ashish 2019; Sarkar et al. 2006). Recovery of waste natural stones in exchange for cement raw materials may cause to significant energy savings processes required for the manufacturing of the final product (İsmail and Ramli 2013; Rana et al. 2015).

The utilization of pozzolans and several admixtures in cementitious composites is a common option for many years (Kara and Arslan 2020). They are employed in concretes, mortars, cements, in the production of structural elements, and combined with other materials such as Portland cement, sand, and lime. Additionally, some al-

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ternative sources may have pozzolanic reactivity. Effects of these materials as pozzolanic materials and for improvement of mechanical performance are depending on their pozzolanic activity. The activity can be identified as the reaction performance of SiO₂ available in the potential material reacting with Ca(OH)₂, resulting in secondary C-S-H gels. Andesite powder is a SiO₂-rich waste, and so it may have pozzolanic activity. Despite all the work about the application fields and natural abundance of andesite, its use as natural pozzolan and its pozzolanic activity in mortar or concrete are not well established. (Hamidi et al. 2013; Davraz et al. 2018)

Sulfate attack is one of the most detrimental effects on the endurance of building materials. After the sulfate exposure, expansion, cracking, and spalling are observed on the cement-based materials (Chindaprasirt et al. 2007; Collepardi 2003; Sancak and Özkan 2015) Sulfate attack occurs in cement-based materials when the materials are in touch with a source of sulfate ions, which can be rainwater, soil or groundwater. Sulfate attack manifests itself by spalling and cracking of concrete caused by dilatation and decrease in mechanical performance. The durability of cement-based materials to sulfate effect is influenced by various factors, such as water absorption and permeability. (Shanahan and Zayed 2007; Sancak and Özkan 2015).

Durability to acid attack is an important environmental effect for building materials employed in structural components. Acid attack is generally originated from several industrial processes and other related applications (Selim et al. 2020). It has been stated that hydrated and unhydrated cement phases are decomposed after subjecting to acid attack. Besides, the damage of attack depends primarily on concentration of acid as well as duration of the attack. Acid effect mechanism includes generally pore expanding bringing about porosity and hence a decline in strength of the exposed building material (Selim et al. 2020; Chi and Stegeman 2000; Beddoe and Dorner 2005; Çelikten 2021).

There are many studies performed regarding substituting marble dusts for cement in cement-based composites. (Yamanel et al. 2019; Ashish 2019; Rana et al. 2015; Selim et al. 2020; Singh et al. 2017; Rashwan et al. 2020; Ghorbani et al. 2020; Khodabakhshian et al. 2018; Zhang et al. 2020, Khyaliya et al. 2017; Ergün 2011). There is a variation in the durability and mechanical performance towards the marble dust incorporation. Literature review has identified a few previous studies on the use of andesite powder in mortar/concrete manufacture (Hamidi et al. 2013; Davraz et al. 2018; Sariisik et al. 2011). Pozzolanic effect of mortar containing andesite powder is reported but the literature on this issue is generally quite limited. Besides, the evaluation of the durability properties of the andesite powder incorporated cementitious composites and comparison of their performance with the plain composites is important.

Acid and sulfate attack on marble powder-based mortars or other cement-based composites has previously been evaluated using mixes modified with waste marble powder. In order to encourage the recovery of waste andesite, this research aimed to determine and compare the resistance of globally studied marble powder-modified mixes with waste andesite-modified mixes under the effect of chemical attacks of acid (HCl) and sulfate (MgSO₄ and Na₂SO₄) solutions. Use of waste natural stones powder in place of cement are carried out in literature. But the usage of andesite powder as substitution material in cementitious composites did not take enough attention; therefore, more study on the issue may attract recycling andesite powder in the cementitious composites which could also help for protection of natural resources. In this study, the blended cements manufactured by using the waste andesite and marble powder were subjected to acid and sulphate solution and the mortars chemical resistance were investigated. Compressive strength, flexural strength, hydrochloric acid resistance, sodium sulfate and magnesium sulfate resistance of cement mortar containing andesite and marble powder were investigated.

2. Materials

2.1. Cement

Ordinary Portland cement produced by Eskişehir Cement Factory of grade CEM I 42.5 R in compliance with TS EN 197-1 (2012) was used in the mortars. The density of the cement was 3.13 g/cm³. X-ray fluorescence results of cement is given in Table 1.

2.2. Andesite and marble powder

Waste andesite powder (AP) and waste marble powder (MP) were employed in the mortar mixtures. The powders were taken from a quarry in Afyon city of Turkey. The density of AP and MP were 2.68 and 2.34 g/cm³, respectively. Before using AP and MP in mortar mixtures, they were sieved from 125 µm sieve and dried at 105°C. The chemical oxide compositions of AP and MP are given in Table 1. Sieve analysis of AP and MP are presented in Table 2.

2.3. Sand

Standard sand (Rilem Cembureau) defined in TS EN 196-1 (TS EN 196-1 2009) was employed for mortar manufacture. The water absorption rate and dry specific gravity of the sand were 0.57% and 2.63, respectively. Sieve analysis of standard sand is given in Table 3.

Table 1. Chemical oxide compositions of cement, andesite and marble powder.

Oxide Content, %	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	K ₂ O	Na ₂ O	MgO	LOI
Cement (PC)	62.87	19.91	5.31	3.23	3.27	0.64	0.29	1.88	2.6
Andesite Powder (AP)	6.32	57.82	19.24	5.87	0.22	2.88	3.14	1.87	1.8
Marble Powder (MP)	58.41	4.57	0.59	0.24	0.17	1.1	0.27	4.56	30.0

Table 2. Sieve analysis of andesite and marble powder.

Sieve size, mm	0.125-0.090	0.090-0.062	0.062-0.012	0.012-0
AP, %	12	29	54	5
MP, %	8	24	61	7

Table 3. Sieve analysis of sand.

Sieve size, mm	2.00	1.60	1.00	0.50	0.16	0.08
Remaining on Sieve, %	0.0	3.2	32.6	68.8	87.2	99.6

3. Methodology

The andesite powder (AP) and marble powder (MP) were utilized in the mortar mixtures as substitution material by the Portland Cement (PC). In preparation of binder paste, AP and MP were employed as a substitution material of cement in 0%, 5%, 10%, 15% and 20% proportion by mass. The proportion of water (W) to total binder content (W/AP+PC) or (W/MP+PC) ratio was 0.5 and sand to total cementitious material proportion of content was 3. The amounts of materials used for the production of mortar mixtures for three-cell mortar mold (4x4x16 cm) are illustrated in Table 4. As seen on the table, the mortar mixes were coded as the substitution of cement respectively. Moreover, the mixing process of the mortars included the following steps. Water and powder binder were poured to the case of Hobart mixer and run at low running rate of 140 rpm for 30 sec. After that, the sand was poured in the bowl in 30 sec. Then, the mixture was mixed at high running rate 30 sec. After high running rate, the mixture was kept waiting at stopped condition for 90 sec. Then, the mixture was mixed at high running rate of 280 rpm for 60 sec. Finally, the mixture was casted into 4x4x16 cm molds after waiting 15 sec. according the TS EN 196-1. After that, the mixes were kept at standard curing conditions until the day of testing. After curing process, mortar specimens with the dimensions of 4x4x16 cm were extracted from the molds to employ the several tests. Three specimens were used for each test and the final results calculated from averaging of results of three specimens.

Firstly, the unit weight values of the mortars were determined in saturated dry surface conditions. Flexural strength (FS) test was conducted on complying with TS EN 1015-11 (TS EN 1015-11:2000) standard, using three-point loading assembly on prismatic specimens with 100 mm span distance between supports. After the FS test, two samples of broken prisms were used to measure the compressive strength (CS) (4x4 cm), complying with TS EN 1015-11. Average of three prismatic samples results were taken as FS value, and average of six samples was taken as CS value. In addition, 6 specimens for each mix were exposed to 5% MgSO₄, 5% Na₂SO₄ and HCl (pH=2) solutions for 90 and 180 days, separately. The FS and CS tests were done on these specimens before and after exposure to the solutions. 56 days aged specimens were used for the FS and CS tests before the immersion to determine the pozzolanic effects of the AP and MP.

Table 4. Amounts of materials used in the mortars (g).

Mix	PC	MP	AP	Water	Sand
PC	450.0	-	-	225	1350
MP5	427.5	22.5	-	225	1350
MP10	405.0	45.0	-	225	1350
MP15	382.5	67.5	-	225	1350
MP20	360.0	90.0	-	225	1350
AP5	427.5	-	22.5	225	1350
AP10	405.0	-	45.0	225	1350
AP15	382.5	-	67.5	225	1350
AP20	360.0	-	90.0	225	1350

4. Results and Discussion

4.1. Unit weight

Unit weights of mortars are illustrated in Table 5. The average unit weight value of PC mortars was 2.09. The unit weights of AP and MP incorporated mortars were ranged between 2.13 g/cm³ and 2.21 g/cm³ and between 2.11 g/cm³ and 2.18 g/cm³ before immersion to acid or sulfate solutions, respectively. The results show that the hardened average unit weights of AP and MP incorporated mortars were up to 3% and 4% higher than the unit weights of PC mortars before immersion to solutions, respectively. The average unit weight of the MP incorporated mortars increased to about 2% and 3% after immersion of the HCl solution for 90 and 180 days, respectively. Due to the difference in the concentration of the HCl, Na₂SO₄ and MgSO₄ solutions and the water in the mortar, the unit weights of the mortars were increased up to 4.2%, 6.1% and 7% after immersion of the solutions, respectively. The increase in the unit weights of the mortars with the solution immersion was compatible with the density of the acid or sulfate solutions.

4.2. Flexural strength

The final flexural strength (FS) results in the mortars are plotted in Figs. 1, 2 and 3. From the results, it could be observed that both powder type and substitution level of cement had important impacts on the final FS development of the mortars. The FS values of mortar specimens after acid solution exposure were compared rela-

tive to the FS values of unexposed specimens. The FS values of AP and MP incorporated mortars after exposure to 90 and 180 days HCl (pH=2%) solutions are illustrated in Fig. 1. After the subjection of HCl solutions for 90 and 180 days, the MP incorporated mortars exhibited the lowest FS values. After the immersion HCl solution for 90 and 180 days the FS values of MP15 mortars were

decreased to almost 4.8% and 17.9%, respectively. Minimum FS reductions in all the mortars were determined for AP15 coded mortars. After the immersion for 90 and 180 days, the FS values of AP15 mortars were decreased to almost 0.3% and 6.1%, respectively. It's concluded that the AP containing mortars performed better against HCl acid attack than the other mortars.

Table 5. Unit weight values of the mortars (g/cm³).

Mortar mixtures	Before immersion	HCl 90d	HCl 180d	Na ₂ SO ₄ 90d	Na ₂ SO ₄ 180d	MgSO ₄ 90d	MgSO ₄ 180d
PC	2.09	2.16	2.13	2.18	2.21	2.17	2.19
MP5	2.11	2.21	2.19	2.14	2.16	2.21	2.22
MP10	2.15	2.21	2.18	2.18	2.19	2.18	2.17
MP15	2.17	2.24	2.22	2.24	2.25	2.20	2.21
MP20	2.18	2.19	2.20	2.25	2.27	2.24	2.25
AP5	2.13	2.23	2.22	2.24	2.26	2.27	2.28
AP10	2.19	2.26	2.21	2.28	2.30	2.28	2.29
AP15	2.17	2.24	2.20	2.25	2.26	2.28	2.31
AP20	2.21	2.23	2.23	2.33	2.32	2.31	2.31

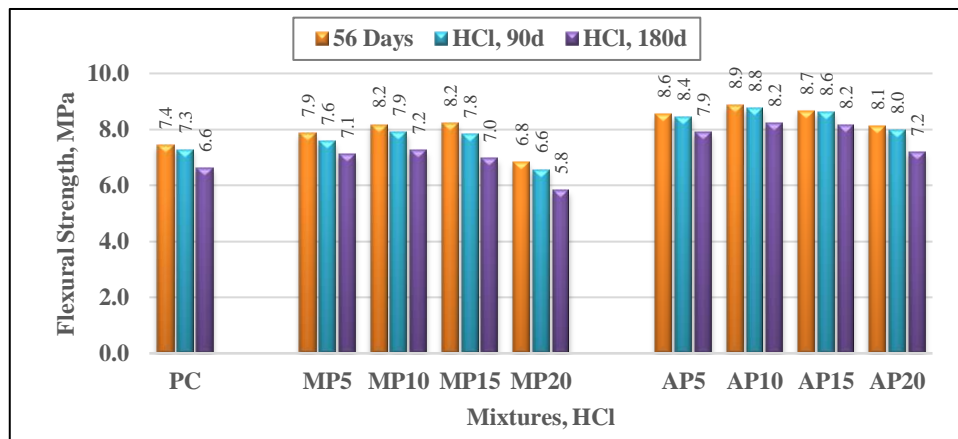


Fig. 1. Flexural strength of mortars exposed to HCl.

The final FS values of AP and MP incorporated mortars mixtures after subjection to 5% Na₂SO₄ solution for 90 days were in the ranges of 6.9-8.2 MPa and 8.1-9.1 MPa, respectively (Fig. 2). The final FS values of AP and MP incorporated mortars after immersion for 180 days were in the ranges of 6.6-8.0 MPa and 8.1-9.0 MPa, respectively. The results indicated that relative to PC mortar mixture, FS increased across all the periods up to 10% AP and MP ratios. FSs of mortars were enhanced with the employment of MP/AP partially in place of cement with respect to PC mortar. When exposure to Na₂SO₄ time increases from 90 days to 180 days, FSs of all the mortars decreased due to degradation of cement paste formation. Maximum FS loss was about 5.8% in MP15 mortars subjected to Na₂SO₄ solution for 180d.

The FS results of the mortars subjected to 5% MgSO₄ solution are represented in Fig. 3. The sulfate resistance of the mortars was considered with respect to difference in FS of specimens after immersion to 5% MgSO₄ solution for 90 and 180 days, separately. After the immersion

for 90 and 180 days, the FS values of PC mortars were decreased to almost 0.5% and 3.0 %, respectively. FS values of MP mortars decreased between 2% and 3.5% after subjecting 5% MgSO₄ solution for 90 days. At 180 days' immersion, the strength losses increased and the strength loss of MP-containing mortars was between 4% and 13%. As for the mortars containing andesite, lower strength values compared to PC mortars under the influence of MgSO₄ for 90 days were only seen in mortars with code AP20. After immersion of 180 days, FS loss was observed between 1.8% and 5.9% in other mortars except AP15. FS loss was not observed in AP15 coded mortars under the influence of MgSO₄ for 90 or 180 days. In general, FS values of AP modified mortars better than MP modified mortars under the 5% MgSO₄ attack for 90 and 180 days. The better performance of the AP-incorporated mortars in the acid and sulfate solutions can be attributed to the pozzolanic activity of the AP. The pozzolanic activity of the AP was also reported in a previous work (Hamidi et al. 2013; Davraz et al. 2018).

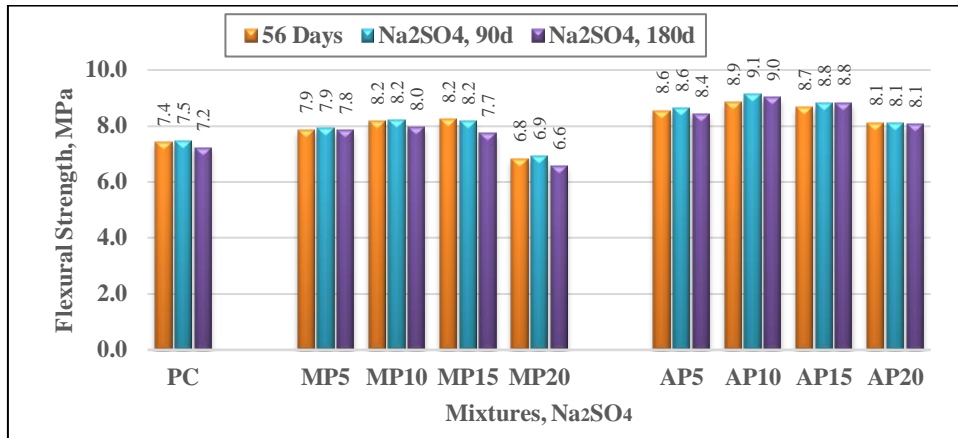


Fig. 2. Flexural strength of mortars exposed to Na_2SO_4 .

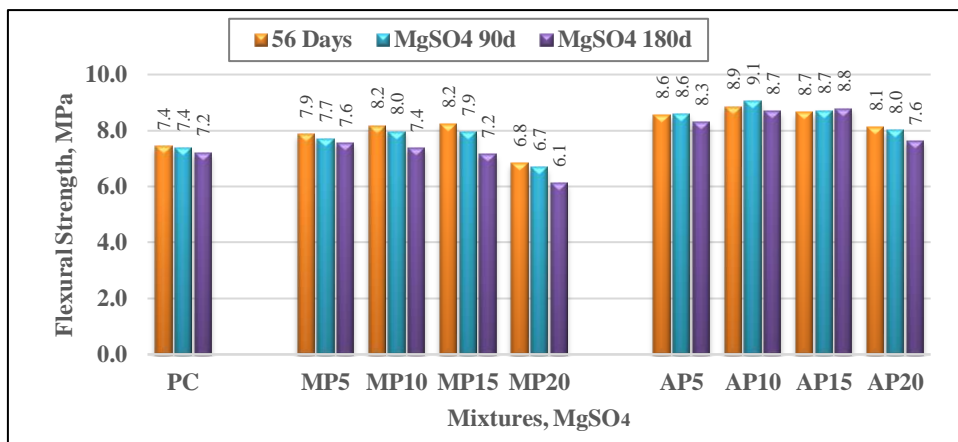


Fig. 3. Flexural strength of mortars exposed to MgSO_4 .

4.3. Compressive strength

The compressive strength (CS) values of mortars after exposure to HCl , Na_2SO_4 and MgSO_4 solutions are presented in Figs. 4, 5 and 6, respectively. The highest CS values were obtained from the AP10-coded mortars before exposure to the solutions. Besides, except for the MP20, the CSs of all the AP or MP incorporated mortars had higher CS than the PC mortar. The positive effect of AP (Hamidi et al. 2013; Davraz et al. 2018) and MP (Yaman et al. 2019; Ashish 2019) on the CS of the Portland cement-based mortars was also reported in previous works. The acid durability of the mortars was determined with respect to change in CS of mortar samples after immersion to HCl solution for 90 and 180 days, separately. Fig. 4 illustrates the initial and final CS results of the mortars after exposure to the HCl solution. The CS results of PC mortars after 90- and 180-days immersion in 5% HCl acid solution were 46.4 and 40.3 MPa, respectively. Initial CS value of PC mortar was 49.3 before immersion to solution. The highest CS values were observed on the MP incorporated mortars for MP5 as 48.6–45.0 MPa and on the AP modified mortars for AP10 as 58.9–52.2 MPa after subjection for 90 and 180 days', respectively. Minimum CS loss was about 10.6% in AP5 mortars after exposure to HCl -180d. Selim et al. (2020) studied the sulfuric and acetic acid resistances of marble dust incorporated Portland cement pastes. Their results

showed that 5% replacement of MP by cement improved the acid resistance of pastes. However, replacement of marble dust by Portland cement at high levels of 15% and 20% decreased the acid resistance of pastes, significantly. These findings are compatible with this present work. The worse acid resistance of MP15 and MP20 mortars with respect to the PC and MP5 mortars can be attributed to the filler effect of MP and its low pozzolanic activity.

The residual CS of the mortars after immersion to 5% Na_2SO_4 solution are shown in the Fig. 5. The final CS values of MP and AP mortars were in the ranges of 46.4–57 MPa and 56.1–69.4 after subjection to Na_2SO_4 solution for 90 days. After exposure for 180 days, average values of the MP and AP incorporated mortars were decreased between 9.1 MPa and 5.8 MPa in comparison with the values calculated after 90 days' immersion. The maximum final CS values of 57.0 and 69.4 MPa were observed on the mortar mixtures manufactured as the MP10 and AP10 in all the series for 90 days, respectively. The enhanced durability may be attributed to the decline of $\text{Ca}(\text{OH})_2$ (Portlandite) formed as a result of the hydration of cement because of the decrement in cement content by substitution. Therefore, the amount of gypsum occurred from the reaction between sulfates and the Portlandite declined, and consequently the ettringite formation, the reason for volume deterioration and expansion, is controlled (Rashwan et al. 2020). The highest CS

value of 61.2 MPa was achieved on the AP10 mortars after subjection to Na₂SO₄ solution for 180 days. Besides, increase in the waste powder ratio had negative influence on the final CS of the mortar mixtures as reported for the results in sulfate environment after %10 ratio. The lowest residual final CS of 39.5 MPa was calculated for the MP20 after subjection to Na₂SO₄ solution for 180 days.

The initial and final CS values of the mortars after subjection in 5% MgSO₄ solution for 90 and 180 days are illustrated in the Fig. 6. While the lowest final CS of 36.4 MPa was seen on the MP20 mortars after immersion to

the solution for 180 days, the highest CS of 62.9 MPa was obtained on the AP10 mortars at same condition. AP mortars had superior performance under the MgSO₄ attack than the PC and MP mortars. Previous studies reported that reducing the content of Ca(OH)₂ in Portland-cement-based composites by incorporating various pozzolans can improve the MgSO₄ durability of these composites and reduce softening of C-S-H gel and the degree of attack (Zhang et al. 2020). The better durability performance of the AP incorporated mortars indicated that AP had considerable pozzolanic activity in addition to its filler effect.

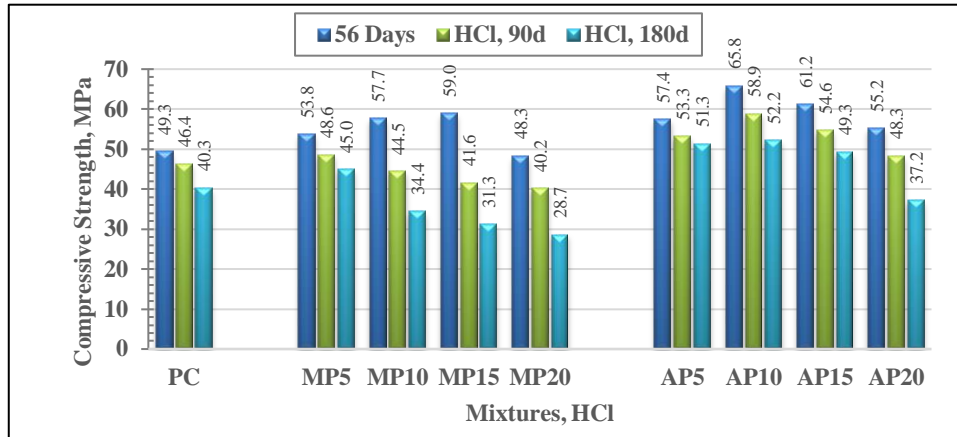


Fig. 4. Compressive strength of mortars exposed to HCl.

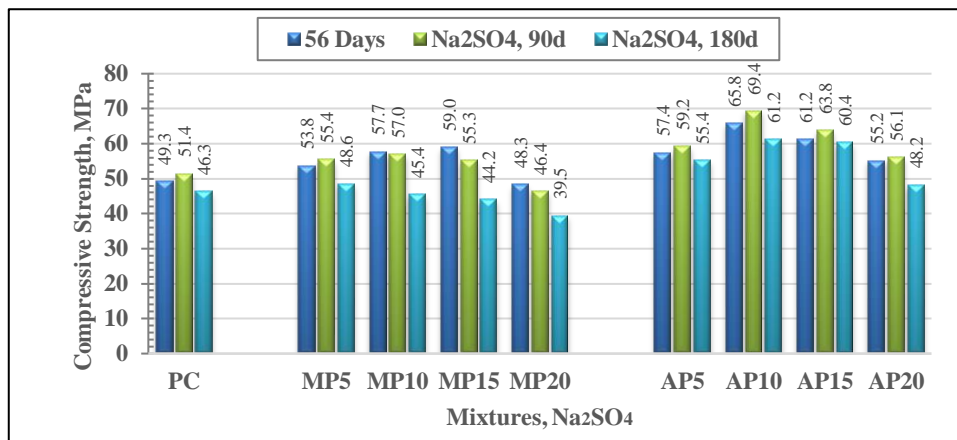


Fig. 5. Compressive strength of mortars exposed to Na₂SO₄.

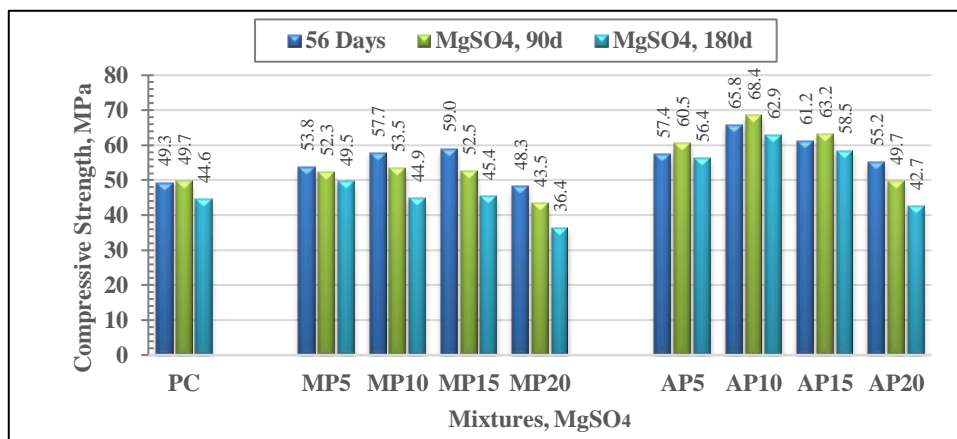


Fig. 6. Compressive strength of mortars exposed to MgSO₄.

5. Conclusions

- Utilization of AP and MP as substitution material for cement may contribute to reduce CO₂ emission by reducing cement consumption. Thus, it can help to conserve the environment.
- The most suitable replacement ratios for concretes containing MP and AP exposed to environmental conditions were determined as 5% and 10%, respectively.
- The strength development of mortar was affected by the level of cement replacement with AP. AP content greater than or equal to 10% is beneficial to the compressive strength of mortar, whereas the flexural strength significantly increases up to an AP content of 15% and begins to decrease at an AP content of 20%.
- The substitution of AP with PC up to 10% enhanced the acid and sulfate durability of the mortars, significantly.
- Substitution of MP by PC up to 15% increased the mechanical properties of mortars at ambient conditions. However, incorporation of MP in PC mortars had negative effect on the durability properties of the mortars. These states indicated that MP had only filler effect on the mortars with very low pozzolanic effect.
- HCl solution had more detrimental influence on the mortars than the sulfate solutions. The CS loss of mortars was in the range of 18% and 47% after immersion of 180d in HCl solution. The CS losses of mortars was in the ranges of 3-25% and 2-25% after immersion of 180d in Na₂SO₄ and MgSO₄ solutions, respectively.

The utilization of AP up to 20% substitution for cement is recommended for mortar production in respect of waste elimination. Besides, use of AP by cement is recommended in mortars subjected to hazardous environmental impacts.

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Conflict of Interest

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