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Enhancement of Sulfate Resistance in Dune Sand Mortars through Polypropylene Fiber Incorporation

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Abstract

This study investigates the impact of different polypropylene fiber (PPF) dosages (0.1% and 0.15%) and lengths (12 mm and 18 mm) on dune mortar (DM) modified with river sand (RS) at a replacement ratio of 50%. The main objective is to evaluate the durability of this fiber-reinforced mortar in a sulfate-rich environment. Compressive strength was assessed at 28, 60, and 180 days, along with mass loss and strength degradation analyses. Microstructural characteristics were examined by scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX). while porosity (*P*) and sorptivity tests were performed to evaluate permeability. The results reveal that PPF reinforcement significantly enhances sulfate resistance, reducing deterioration rates in mass and compressive strength by up to 46% and 40.87%, respectively. Additionally, water absorption and sorptivity coefficient (*S*) decreased to 83.91% and 86.36%, respectively, compared with the control. The synergy between modified sand content and PPF reinforcement demonstrates potential for improving sulfate durability, making it a viable solution for concrete applications in aggressive environments.

Keywords

polypropylene fibers, dune mortar, sulfate resistance, microstructural analysis, sorptivity

1 Introduction

Recent studies have explored the use of dune sand (DS) in concrete production, showing promising results in terms of strength and durability. Partial replacement of river sand (RS) with DS (up to 40%) can be achieved without negative effects on concrete properties [1–3]. Sulfuric acid attack significantly reduces concrete durability, particularly in sewer systems. Research has shown that mineral admixtures can enhance the resistance of concrete to sulfuric acid and sulfate attacks [4]. The efficacy of these admixtures is influenced by several factors, such as cement content, water-cement ratio, and the proportion of coarse aggregates [5].

Polypropylene fiber-reinforced concrete (PFRC) demonstrates improved resistance to sulfuric acid attack compared to plain concrete. The addition of polypropylene fibers (PPFs), particularly a volume fraction of 1.04%, decreases the negative impacts of sulfuric acid on the compressive strength of concrete, with higher temperatures and reduced pH leading to more severe degradation [6]. PPFs reinforced polymer concrete exhibits excellent resistance to sulfuric acid attack, with no changes in volume or weight observed after 11 weeks of exposure to 5% and 10% sulfuric acid solutions [7]. The inclusion of hybrid fibers, including polypropylene, in self-compacting concrete (SCC) subjected to sulfuric acid attack was also investigated, revealing microstructural changes and gypsum formation [8]. PPFs added to concrete with iron tailings sand improved its resistance to sodium sulfate attack, with reduced pore development, crack formation, and sulfate ion diffusion [9]. Multiscale PFRC exhibits the highest compressive strength and lowest sulfate ion penetration after exposure to sodium sulfate solution [10]. Overall, these findings highlight the potential of PPFs to improve in enhancing concrete's the durability of concrete against sulfuric acid attack. Concrete is a widely used construction material, but its durability can be significantly compromised by aggressive environmental conditions, particularly acid attacks. This study purposes to examine the use of DS in concrete, incorporated at a dosage of 50%

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dosage, and to analyze the impact of PPFs on the durability of mortars exposed to a sulfuric environment containing 5% H_2SO_4. A comparative study was conducted between a reference mortar without additives and mortars containing PP fibers at varying dosages. The specimens were monitored through a rigorous methodology to highlight the physical, mechanical, and chemical aspects of mortar deterioration. Key analyses included compressive strength tests, mass variation measurements, scanning electron microscopy (SEM) remarks of undamaged and degraded sections, and X-ray diffraction (XRD) investigations to detect microstructural changes.

2 Experimental program

2.1 Materials

The cementitious material used for the production of DS mortars was ordinary Portland cement CEM II/A-L 42.5N, in compliance with EN 196-1:2016 standard [11]. This cement had a specific gravity of 3.07g/cm^3, the chemical characteristics are presented in Table 1 [12].

This study utilized two types of sand: DS and RS. DS was obtained from Ain El-Beida in the Ouargla region and is characterized by round and isometric grains. On the other hand, RS extracted from the Hassi Sayah (Ouargla), has grains irregular shape and variable sizes. The granulometric analysis of the sands (as per the P 18-560 standard [13]) indicate that DS does not meet the granulometric requirements for ordinary concrete, necessitating a correction by adding 50% RS (Fig. 1). Table 2 provides a summary of the physical and chemical characteristics of the sands.

Table 1 Chemical characteristics of cement used [12]

Element	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	LOI
Unit (%)	18.86	4.38	3.12	62.21	1.23	2.7	0.72	6.08

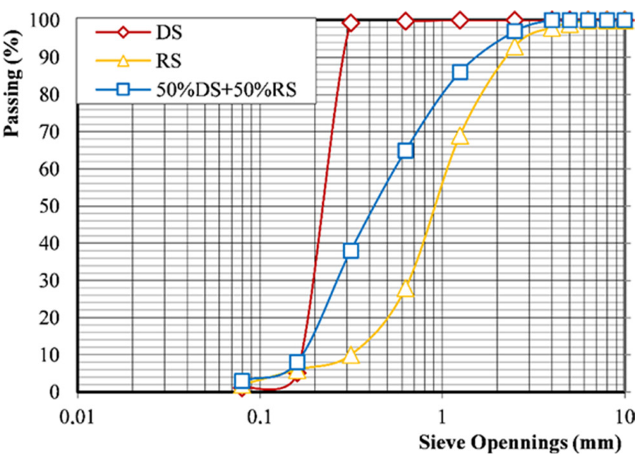


Fig. 1 Granulometric analysis of the sands

Table 2 Physical and chemical characteristics of sands

Characteristic	Nomination	Sand type	
		DS	RS
Physical	Apparent density (g/cm^3)	1.45	1.61
	Absolute density (g/cm^3)	2.67	2.64
	Sand equivalent (%)	93.11	74.28
	Water absorption (%)	1.04	0.56
	Fineness modulus (%)	0.95	2.95
	Porosity (*P*) (%)	45.69	38.31
Chemical	SiO_2	89.71	90.48
	Al_2O_3	0.52	0.51
	Fe_2O_3	0.78	0.91
	CaO	0.41	0.04
	MgO	0.20	0.27
	SO_3	0.47	0.31
	K_2O	0.24	0.24
	Na_2O	0.10	0.08
	P_2O_5	0.01	0.01

The PPFs utilized are FIBERTEK PP fibers, have standard lengths of 18 mm (F1) and 12 mm (F2). These fibers feature a specific gravity of 0.91 g/cm^3, a diameter of 32 µm, a melting point of 160 °C, a tensile strength of 450 MPa, an elongation at failure of 40%, and a modulus of elasticity of 3700 MPa for F1. F2 fibers share similar characteristics, except for their shorter length of 12 mm [14]. The water-reducing plasticizer MEDAFLUID 40 is used to improve concrete mixing, particularly when fibers are included. It has a density of 1.19, a pH between 8 and 9, and a chlorine content of less than 1 g/L [15].

2.2 Method

2.2.1 Formulation and preparation of specimens

For the composition of the control dune mortar (DM) and corrected dune mortar (DRM), made respectively with DS and a mix of 50% DS and 50% RS, we adopted a cement-to-sand ratio corresponding to standardized mortar in compliance with the EN 196-1:2016 standard [11], which specifies one part cement to three parts sand. Regarding the water dosage, we conducted a workability test to determine the appropriate amount of water and admixture required to achieve a workable mortar. Based on this test, the water-to-cement (W/C) ratio for mortar (DM) and corrected mortar (DRM) was 0.58 and 0.47, respectively. From the control mortar mix, mortars incorporating PPFs were prepared at different fiber contents of 0.10% and 0.15% of the volume. Two types of fibers were used: F1 (18 mm in length) and F2 (12 mm in length). The fiber-reinforced mortars were designated as DM_0.10F1 for DM mortar containing 0.10% of F1 fibers, and so on. The detailed proportions of the various mixes are presented in Table 3.

2.2.2 Test methods

All tests cited below were conducted using three specimens for each case, with dimensions of 4 × 4 × 16 cm^3.

Durability

Durability reflects the concrete's ability to resist physical and chemical degradation. It was assessed through porosity and sorptivity tests:

1. Porosity:
   The water-accessible porosity (P) was measured on specimens of 4 × 16 × 16 cm^3 following the NF P18-459:2022 standard [16]. The procedure began with immersing the samples in a water tank for at least 48 h to ensure they reached a consistent wet weight (W_{wet}). Afterward, the specimens were dried in an oven at 105 ± 3 °C for a minimum of 72 h until a stable dry weight (W_{dry}) was obtained. Hydrostatic weighing (W_{hyd}) was then conducted to measure the mass of the specimens in water. P was calculated using Eq. (1):

$$P(\%) = \left[\left(W_{wet} - W_{dry}\right) / \left(W_{wet} - W_{hyd}\right) \right] \times 100. \quad (1)$$

2. Sorptivity test:
   Sorptivity test evaluates the ability of a porous material to absorb water through capillary action. Specimens are dried in an oven at 105 °C until a constant mass is achieved. The bottom face is then immersed in water to a depth of 2–5 mm, and the mass is recorded at regular intervals (e.g., 5, 10, 15, 30 min) over a specified period. Sorptivity coefficient (S) is calculated from the slope of the linear relationship between the cumulative water absorbed per unit area and the square root of time.

Sulfuric acid attack

After 28 days of water curing, specimens were weighed to determine their initial mass and then immersed in solutions containing 5% H_2SO_4 added to the water volume. Chemical resistance was evaluated by measuring the mass of the specimens in accordance with the ASTM C267-97 standard [17]. The specimens were cleaned three times with fresh water to remove deteriorated concrete, dried for 30 min, and weighed again. This procedure was repeated after 7, 14, 21, 28, 46, 60, 90, 120, 150 and 180 days of immersion. The solutions were renewed every 14 days, based on pH variations.

Compressive strength

Specimens were preserved in water for 28 days during the curing period. After curing, they were immersed in a sulfuric environment for 28, 60, and 180 days. At the end of each exposure period, the specimens were subjected to compressive strength testing in accordance with the NF EN 12390-3 standard [18]. The load was applied continuously until the specimens ruptured.

Scanning electron microscopy (SEM)

The SEM analysis was conducted using a field emission instrument (FEI Quanta FEG 250, Hillsboro, OR, USA) with an accelerating voltage ranging from 200 V to 30 kV and a magnification range of 30X to 300,000X. The SEM was equipped with an energy-dispersive spectrometer (EDS) from EDAX (Energy Dispersive Analysis of X-rays), enabling the visualization of grain structures on polished sections or pellets, performing targeted chemical analyses of individual mineral phases, and quantifying their proportions through image analysis.

Table 3 Mortars mix (1 m^3)

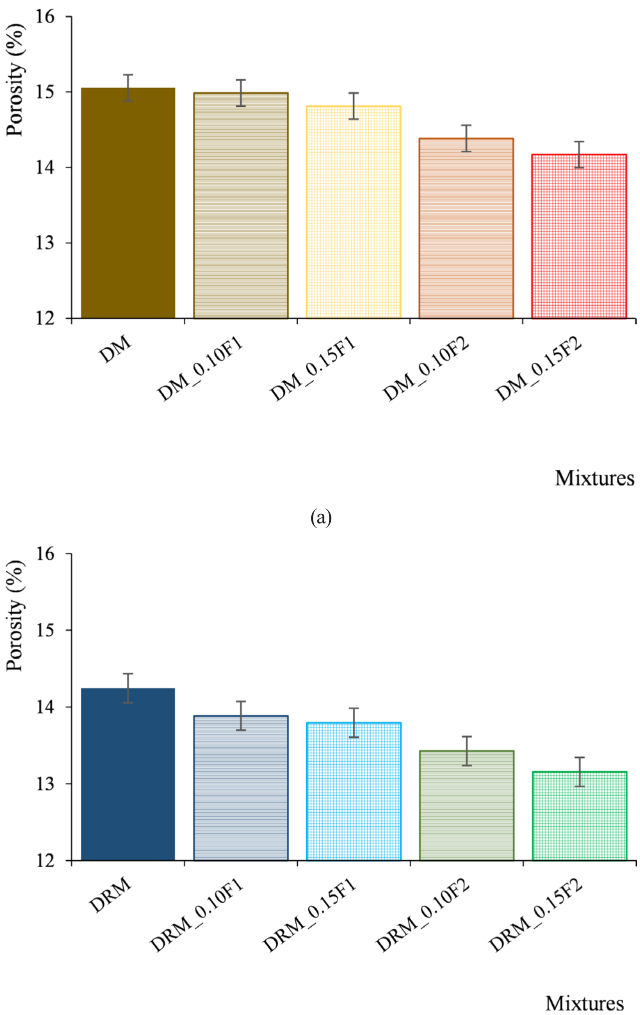
Mixtures	Sand (kg)		Cement (kg)	Water (L)	Fibers (kg)	Plasticizer (kg)	W/C
	DS	RS					
DM	1388.7	0			0	0	
DM_0.10F1	1386	0			1	1.7	
DM_0.15F1	1384.6	0	462.9	268.5	1.5	2.5	0.58
DM_0.10F2	1386	0			1	1.7	
DM_0.15F2	1384.6	0			1.5	2.5	
DRM	711.4	711.4			0	0	
DRM_0.10F1	710.1	710.1			1	1.7	
DRM_0.15F1	709.4	709.4	474.3	222.9	1.5	2.5	0.47
DRM_0.10F2	710.1	710.1			1	1.7	
DRM_0.15F2	709.4	709.4			1.5	2.5	

3 Results and discussion

3.1 Durability

3.1.1 Porosity

Figs. 2 (a) and (b) present the *P* results of DM and DRM samples. The data show that the *P* of DRM, with or without fibers, is lower than that of DM. This lower *P* serves as an effective barrier against harmful agents, highlighting the beneficial effect of granulometric correction of DS in the DRM mix. This correction reduces the number of large pores, leading to a decrease in *P* and an increase in the internal structural compactness of the concrete [3]. For fiber-reinforced mixtures, DRM exhibits reduced *P* compared to DM. However, the variation in *P* between mixtures with and without fibers remains relatively small. This could be attributed to better fiber distribution and enhanced interaction with the cement paste. From Fig. 2, it can also be observed that the length of PPFs significantly influences the concrete's *P*. Short fibers show a more positive effect. Specifically, mortars reinforced with 0.15% short fibers (F2) recorded the greatest reduction in *P*, reaching 5.89% for DM and 7.66% for DRM. This effect can be explained by the high specific surface area of short fibers, which improves their contact with the cement paste and thus reduces overall *P*. These results are confirmed by studies conducted by Meziani et al. [3] and Pliya [19]. However, several other studies Hager [20] and Lublóy [21] have reported that incorporating PPFs into ordinary concrete can sometimes result in a slight increase in *P*.

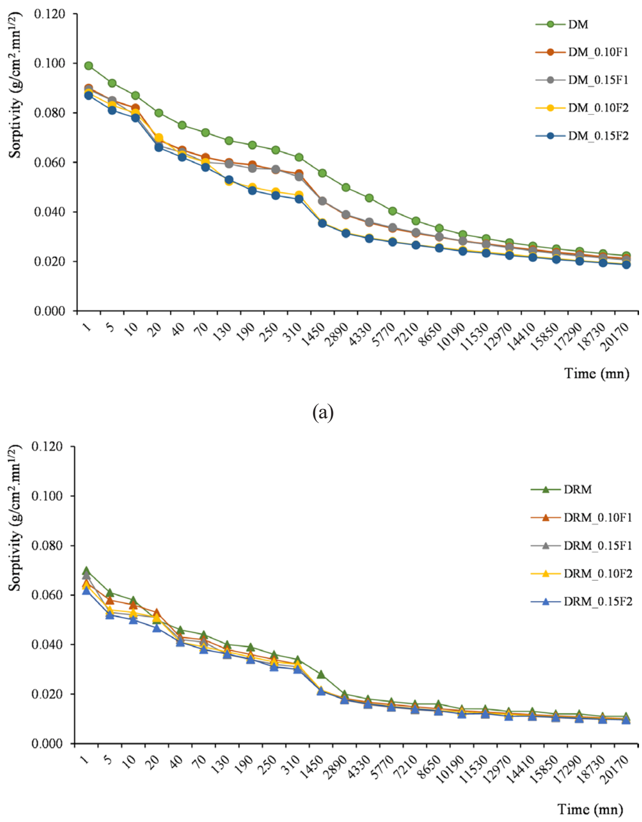


(b)

Fig. 2 Porosity of DMs: (a) DM; (b) DRM

3.1.2 Sorptivity test

The two graphs in Fig. 3 illustrate the sorptivity over time for various DM and DRM mixes, with or without PPFs (F1 or F2). All mixes show a decrease in sorptivity over time, indicating a progressive reduction in water absorption capacity, with DM mortars (without fibers) consistently exhibiting the highest values, suggesting greater *P* and poorer resistance to absorption. The addition of fibers (F1 and F2) leads to a significant reduction in sorptivity compared to DM mortars without fibers, with the DM_0.15F2 mix showing the lowest sorptivity, reaching 0.025 (cm/s$^{1/2}$) after 15,000 min, highlighting the effectiveness of 0.15% short fibers in enhancing resistance to capillary absorption. Short fibers (F2) are more effective than

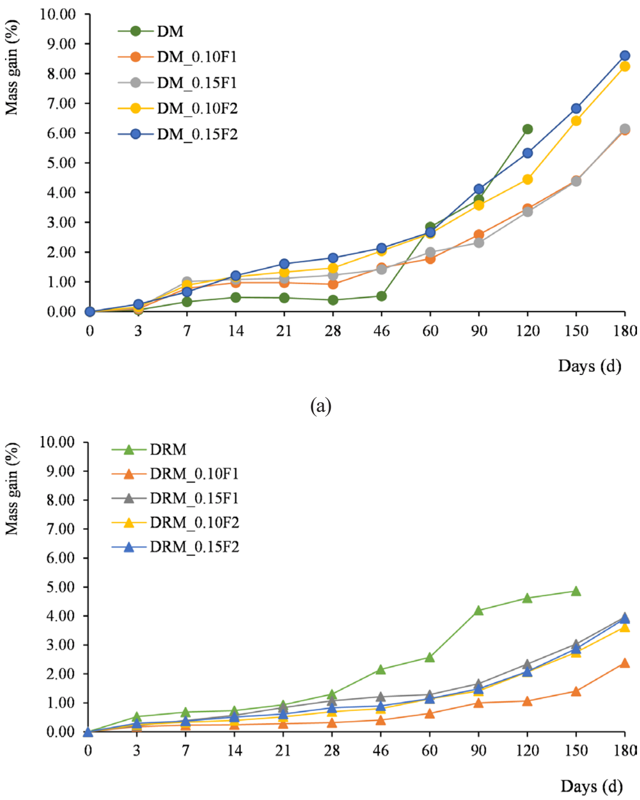


(b)

Fig. 3 Sorptivity for various DM over time: (a) DM; (b) DRM long fibers (F1), as evidenced by the general trend where mixes containing F2 display lower values. DRM mortars also show lower sorptivity than DM mortars, both with and without fibers, indicating that particle size correction reduces interconnected pores and enhances resistance to water penetration; for instance, the DRM without fibers has a sorptivity of 0.012 (cm/s$^{1/2}$) after 15,000 min, which is significantly lower than the values observed for DM. Furthermore, the reduction in sorptivity is more uniform in DRM mortars, suggesting better fiber distribution within the granulometrically corrected matrix, which could be attributed to improved compactness and a more homogeneous internal structure. These results corroborate previous studies showing that fibers can optimizes pore size distribution, which reduces the pathways for water ingress, leading to lower sorptivity values [22, 23]. The particle size correction of DS positively impacts mortar performance by reducing its *P* and increasing its resistance to water absorption.

3.2 Sulfuric acid attack

Fig. 4 (a) and (b) illustrate the mass increase of concrete formulations exposed to a sulfuric environment (5% H_2SO_4), revealing significant trends based on composition and the type of fibers used.



(a)

(b)

Fig. 4 Mass gain for various DM over time: (a) DM, (b) DRM

Formulations with DM exhibit higher mass increases compared to formulations with a mixture of DRM. At 28 days, DM shows a mass increase of up to 0.39%, while DRM_0.10F1 shows 0.32%. At 90 days, DM_0.10F2 reaches 3.57%, whereas DRM formulations maintain mass increases between 1% and 1.4%.

No DM mortar specimens without PPFs remained after 150 days of exposure, as they exhibited significant degradation and damage (Fig. 5).

In contrast, the DRM specimens retained their structural integrity and shape. This improved performance of DRM mortars can be attributed to the presence of RS in the mixture, which significantly enhances resistance to sulfuric acid attacks. RS provides better particle size distribution, reduced chemical reactivity, and higher density, resulting in a more compact matrix with lower permeability. These findings align with Neville [24], who emphasized the role of optimized aggregate combinations in enhancing durability. These properties minimize the penetration of sulfate ions and reduce the formation of expansive degradation products like gypsum ($CaSO_4{\cdot}2H_2O$) and ettringite ($3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O$), which are responsible for cracking, swelling, and structural damage in cementitious materials. After 180 days of sulfate exposure, no mortar specimens without fibers (DM and DRM) could be retrieved intact, as the swelling caused by the sulfate attack led to severe degradation and deformation. Among mortars with fibers, DRM_0.15F2 recorded the highest mass increase at 8.61%, while DRM_0.10F1 showed a significantly lower increase at 2.38%.

The increase in mass observed in all mortars exposed to sulfuric acid is primarily attributed to the formation of

Fig. 5 Degradation and damage of specimens DM after 150 days of exposure

degradation products such as gypsum and ettringite. These compounds accumulate within the porous matrix of the concrete due to chemical reactions between sulfate ions in the acidic solution and the hydrated cement phases, such as portlandite ($Ca(OH)_2$) and calcium silicate hydrate (C–S–H) gel. Studies confirm that the presence of these degradation products leads to an increase in mass, though often accompanied by internal structural degradation [25].

Additionally, the use of PPFs, especially at a concentration of 0.15%, has been shown to significantly mitigate this increase in mass. Shorter fibers (F2) offer enhanced performance in reducing overall mass gain, providing a more durable solution in aggressive environments. According to recent studies, the incorporation of PPFs reduces mass gain by up to 25% at a concentration of 0.15%, as supported by statistical analysis showing a reduction in mass gain from 8.9% to 6.7% over 180 days of exposure in a sulfuric acid solution.

3.3 Compressive strength

Fig. 6 (a) and (b) show that DM concrete is the most vulnerable to acid attack, with compressive strength losses increasing significantly over time, reaching 66.39% after 180 days. This weakness can be explained by the nature of

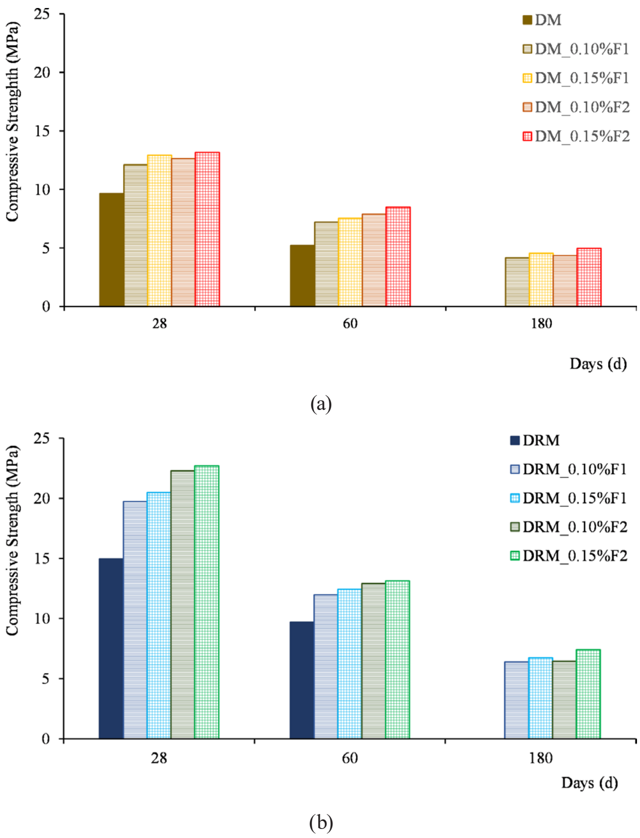


Fig. 6 Compressive strength of mortars at different ages: (a) DM; (b) DRM

DS, often rich in reactive fines, which accelerate chemical degradation in acidic environments [2].

The inclusion of RS in the DRM mixture slightly reduces the losses, with a value of 74.47% at 180 days, likely due to better granulometry and lower chemical reactivity of RS, improving the concrete's structure.

Fig. 7 shows a significant decrease in the compressive strength of mortars exposed to a sulfuric environment (5% H_2SO_4) over time.

At 28 days, the DRM mortars with 0.15% short fibers (12 mm, F2) achieve a maximum strength of 23 MPa,

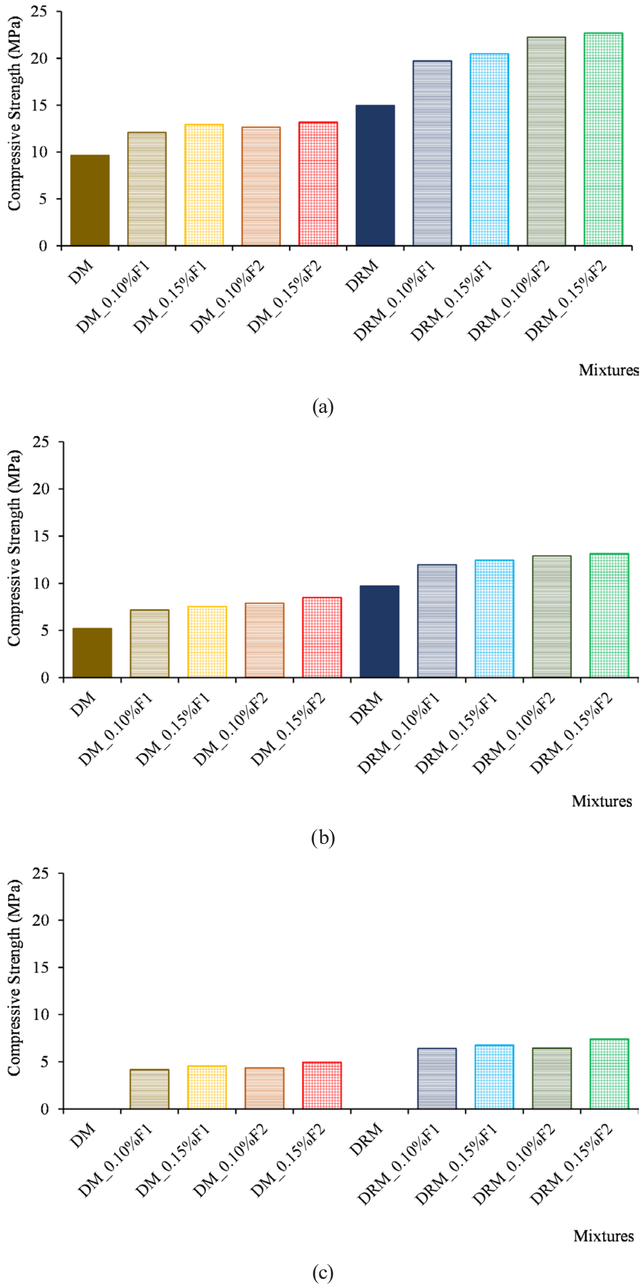


Fig. 7 Influence of PPFs dosage on compressive strength at different ages: (a) 28 days, (b) 60 days, (c) 180 days

while formulations without fibers (DM and DRM) remain around 10 and 15 MPa respectively. At 60 days, a notable decrease is observed: fiber-reinforced formulations maintain a strength of 10–14 MPa, compared to 7–10 MPa for those without fibers. At 180 days, the compressive strength of the fiber-reinforced mortars (DM_015F2 and DRM_015F2) is around 4–8 MPa, confirming their better durability against sulfuric attack.

The improvement due to fibers is explained by their ability to limit crack propagation by acting as a bridge in the matrix, which enhances internal cohesion and reduces the destructive effects of expansion products such as gypsum and ettringite. Short fibers (F2) are particularly effective due to their better dispersion within the matrix, as demonstrated by Mahdavi et al. [6], Banthia and Gupta [26], and Kantarcı [27], who confirmed that PPFs leads to a notable reduction in compressive strength loss when exposed to sulfuric acid, with improvements in durability metrics such as freeze-thaw resistance and sulfate attack resistance.

Additionally, the use of RS in the DRM formulations improves compactness and reduces permeability, limiting the access of sulfate ions to the cement matrix.

3.4 Microstructural analysis tests

Fig. 8 presents the SEM image of a DRM_0.15F2 sample after 180 days of exposure to 5% sulfuric acid. The surface exhibits a highly porous structure, with numerous microcracks and voids visible throughout. Significant decomposition of C–S–H gel into finer particles is evident, along with remnants of calcium hydroxide crystals.

Additionally, gypsum formations are clearly observed covering the surface. The extensive gypsum formation in the surface regions appears to have contributed to the disintegration and spalling of the material's surface (Fig. 9).

The Table 4 presents SEM-EDS analysis of the DRM_0.15F2 specimen exposed to a 5% sulfuric acid (H_2SO_4) environment highlights key chemical modifications and interactions within the matrix under aggressive conditions. Oxygen dominates the elemental composition with the highest mass percentage (51.90%), reflecting the formation of oxides or hydroxides due to the acidic degradation of the cementitious matrix [28]. Silicon, with a significant mass percentage of 15.59%, indicates the presence of silicate-based compounds, likely residual unreacted silica or modified C–S–H) [29]. Calcium, constituting 17.22% by mass, suggests the formation of gypsum ($CaSO_4{\cdot}2H_2O$) or other calcium sulfate hydrates, characteristic of sulfuric acid attack [30]. Carbon, at 5.86% mass percentage,

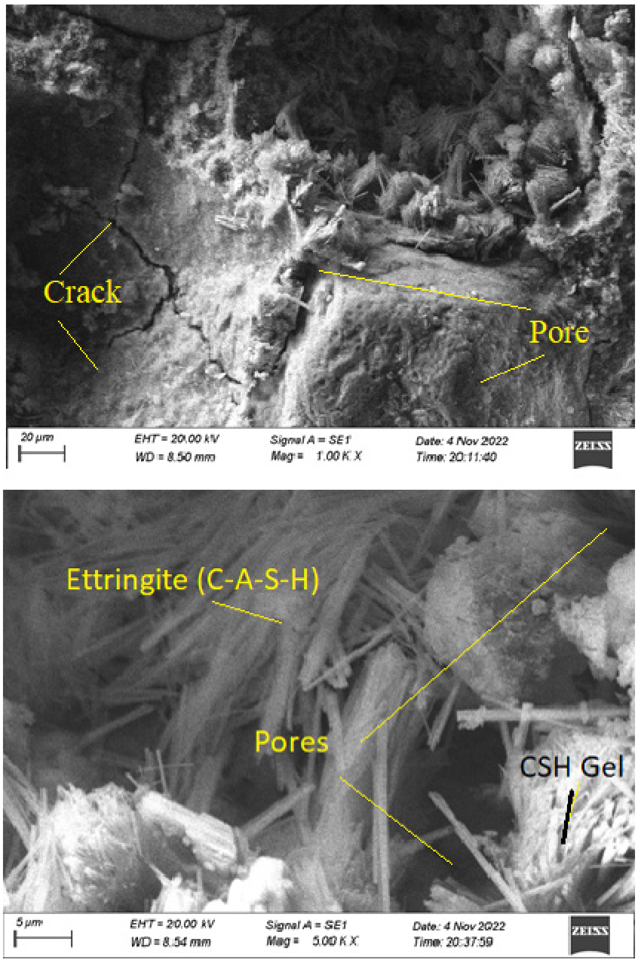


Fig. 8 SEM of DRM_0.15F2 after 180 days of exposure to 5% H_2SO_4

(a) (b)

Fig. 9 DRM_0.15F2 specimens at: (a) 28 days; (b) 60 days

points to potential carbonation, contributions from PPFs, or residual carbonates in the matrix. Minor elements like magnesium (1.18%), aluminum (2.40%), and potassium (0.79%) suggest the presence of dolomitic aggregates, alumino-silicate phases, and alkalis, respectively. Overall,

Table 4 SEM-EDS analysis of the DRM_0.15F2

Element	C	O	Mg	Al	Si	MoL	K	Ca
% by mass	5.86	51.90	1.18	2.40	15.59	5.06	0.79	17.22
% by atomic	9.91	65.84	0.98	1.80	11.26	1.07	0.41	8.72

the results confirm that the sulfuric acid environment altered the matrix by forming gypsum and modifying silicate phases. The inclusion of 0.15% short PPFs likely enhanced durability by mitigating cracking.

4 Conclusions

The study demonstrates that the granulometric correction of DS and the incorporation of PPFs significantly enhance the durability and performance of dune-based mortars under aggressive conditions. Key findings include:

- DRM exhibit reduced *P* and sorptivity compared to uncorrected DM, with the most notable reductions observed in mixes containing 0.15% short PPFs (F2). This improvement is attributed to better particle size distribution, enhanced fiber-matrix interaction, and increased compactness of the concrete.
- Under prolonged exposure to a 5% sulfuric acid solution, DRM mortars retained structural integrity and demonstrated lower mass gain and compressive strength losses than DM mortars. The use of short fibers (F2) further improved acid resistance, mitigating degradation caused by gypsum and ettringite formation.
- Fiber-reinforced mortars showed better retention of compressive strength over 180 days in an acidic environment, with DRM_0.15F2 achieving the highest durability. This enhancement is due to the fibers' ability to bridge cracks and limit the propagation of damage induced by sulfates.
- SEM-EDS analyses confirm that the addition of short PPFs reduces cracking and degradation of the cementitious matrix, even under severe sulfate exposure. The formation of degradation products, such as gypsum, is minimized in DRM mortars, further highlighting their superior durability.
- These findings emphasize the potential of optimized DS mortars, particularly with short PPFs, as a durable material for construction in sulfate-rich environments. Future studies should explore the long-term performance and additional applications of such materials to maximize their engineering potential.

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