



Mechanical and Durability Characteristics of Mortars Reinforced with PET Fibers

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Abstract

Recycling and recovery of waste are today considered a solution for the future in order to respond to the deficit between production and consumption and to protect the environment. The objective of this work is to evaluate the durability characteristics of mortars reinforced with PET fibers and Ground Granulated Blast Furnace Slag (GGBFS) or silica fume as mineral additions. Different mixtures were exposed in the external sulphate attacks and tested for mechanical strength, loss of weight at different ages. The analysis of tests results showed that the mineralogy, morphology and the rate of mineral admixtures are significant factors affecting the durability of mortars. The mortars with silica fume after one year of conservation in a high magnesium sulphate medium (MgSO4) present the lowest damage compared to mortar with GGBFS.

Keywords: Ground granulated blast furnace slag (GGBFS); Sulphate attack; PET fibers; Mechanical strength; Microstructure

Introduction

The broad awareness of ecological, social and economic imperatives, the search for sustainable technologies, the growing problem of waste, environmental legislative standards as well as the depletion of fossil resources are at the origin of the direction of scientific research towards the development of sustainable materials [1]. To ensure the durability of materials, it is not only necessary to take an interest in the methods of formulation and implementation, but also in the environmental conditions. All outdoor environments are considered aggressive for materials with a cementitious matrix [2-4]. The chemical attacks that cementitious matrix materials may encounter are very varied. Due to its porosity and the chemical composition of the interstitial solution, material exchanges can occur and be the cause of a change in the composition of the cement paste. These transport and reaction phenomena take place on the scale of the microstructure of the cement paste. There are two types of attacks: attacks by external sulphates present in the environment of the concrete and internal sulphate attacks for which the sulphates come from the components of the concrete itself.

External sulfate attack is the cause of one of the most aggressive environmental deteriorations affecting the durability of concrete structures. The phenomenon of the attack of external sulfates on concrete has been known since the end of the 19th century when Michaelis observed white deposits in concrete exposed to sea water, which he designated under the name of "bacillus of cement". This phase was later recognized as ettringite [5]. The sulfate attack is associated with the precipitation of secondary sulfate products, an expansion



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Submission: December 15, 2023 Published: February 14, 2024

Volume 5 - Issue 4

How to cite this article: Yasmina Biskri*, Laidi Babouri, Hanane Dob and Ouided Dehas. Mechanical and Durability Characteristics of Mortars Reinforced with PET Fibers. Polymer Sci peer Rev J. 5(4). PSPRJ. 000616 2024. DOI: 10.31031/PSPRJ.2024.05.000616

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significant and chemo-mechanical deterioration (changes in the transport properties of the porosity, cracks, loss of strength and cohesion, etc.). This can lead to the ruin of the material cementitious, in the more or less long-term depending on the attack (nature, content and concentration of sulfates in contact) and the cement used (type and Water/Cement ratio).

Signs of deterioration by the attack of sulphates on the concrete are characterized by swelling due to the formation of expansive products which create internal tensile stresses, which leads in the long term to expansion followed by surface cracking, then an increase in permeability and finally a decohesion of the constituents of the concrete which are responsible for a reduction in the mechanical properties [6,7]. Several studies [8-11] show that there are some parameters which influence this degradation, namely the type and concentration of the attack solution as well as temperature. They proved that the magnesium sulfate solution MgSO₄ is more aggressive than the sodium sulfate solution Na₂SO₄ and the increase in temperature accelerates the attack of these sulfates. The utilizations of by-products in concrete production such as fly ash, bottom ash, slag, silica fume, waste glass, etc. are the solutions to achieve the above objectives [12-14].

Some researchers have reported that the type of cement and mineral additions influence the degradation of cementitious materials, the use of low water/cement ratio and the use of admixtures, such as air entraining or ground Granulated Blast-Furnace Slag (GBFS), would be the most effective treatment for reducing the sulphate-inducing damage [15]. Besides, mineral admixtures have shown to improve the sulphate resistance of cementitious systems due to their pore refinement, grain refinement, C₂A dilution and removal of calcium hydroxide by pozzolanic reaction. Under the sulphate environment, cement paste undergoes deterioration resulting from expansion, spalling and softening [16-19]. The use of compound cements or the addition of granulated slag is essential for improving the durability of concrete structures and has technical, economic and environmental advantages. Blast furnace slag improves the resistance of concrete to sulfates by diluting aluminates, reducing the portlandite content and increasing the compactness of hydrates in relation to the reduction in pore volume [20,21]. This study aims to analyze and evaluate the effect of the attack of magnesium sulfate (MgSO₄) on the physico-mechanical and microstructural degradation of sand concretes based on blast furnace slag or silica fume.

Materials and Method

Materials

Binder: The binders used were Portland cement CEM I 42.5/A as classified by the European Standard EN 197-1 [22] and two mineral admixtures. These were the silica fume according to Standard NF EN 13263-1 [23] and the Algerian blast furnace slag (GGBFS) according to Standard NF EN 15167-1 [24]. The glass content of Algerian slags is greater than 93% and therefore has a relatively slow hydraulic activity. Table 1 describes the various chemical and physical-mechanical properties of cement and mineral admixtures.

Table 1: Chemical and physical-mechanical properties of cement and mineral admixture.

Chemical Composition (%)				
Component (%)	Cement	GGBFS	Silica Fume	
(SiO ₂)	21.91	38.90	99.01	
(Al ₂ O ₃)	5.19	8.17	0.02	
(Fe ₂ O ₃)	2.94	4.15	0.05	
(CaO)	60.41	40.96	0.05	
(MgO)	1.60	9.53	0.01	
(Na ₂ O)	0.16	0.10	0.03	
(K ₂ 0)	-	0.89	0.15	
(SO ₃)	2.19	0.36	0.001	
Cl-	0.02	0.01	0.009	
Physical Properties				
Fineness (cm ² /g)	4120	3800	6700	
Absolute density (g/cm ³)	3.10	2.88	2.24	
Mechanical Properties				
Compressive strength at 28 days (MPa)	44.60	-	-	
Flexural strength at 28 days (MPa)	7.50			

Aggregates: Two different size fractions of 0/2mm fine siliceous sand and 1.25/5mm limestone crushed sand of aggregates

were used. Table 2 describe the physical properties.

Siliceous Sand	Limestone Crushed Sand			
2.53	2.60			
75	80			
1.85	2.70			
0.55	0.5			
	Siliceous Sand 2.53 75 1.85 0.55			

Table 2: Physical properties of sand.

PET fibers: The PET fibers used in this study were provided by the company RET-PLAST, located in the region of Mezloug/Sétif (Algeria), specialized in the recycling of post-consumer PET bottles. The observations of the PET fiber using the optical microscope and the scanning electron microscope are presented in Figures 1a & 1b, respectively. The physical-mechanical characterization of the PET fiber was carried out at the MediFil Company in Hammam Guergour/Sétif, Algeria. The corresponding results are given in Table 3.

Table 3: Physical and mechanical properties of PET fibre.

Properties	Values	
Density at 20 °C	1,16	
Cutting length (mm)	70,00	
Metric Number (Nm)	557,00	
Title (DTex)	18,00	
Size (Denier)	16,16	
Pressley Index (lb/mg)	6,97	
Pressley (Pound/ Pouc ²)	73,00	
Breaking length (gf/Tex)	37,39	
Relative Toughness (gf/ Denier)	4,00	



Figure 1: PET fibers: (a) optical photo and (b) SEM photo.

Sulphate: The sulphate attack solution used for the experiment is a solution of magnesium sulphate ($MgSO_4.7H_2O$) with a concentration of 50g/l according to standard NF P18-837.

Super-plasticizer: The super-plasticizer used is a highly water-reducing super-plasticizer marketed by the company Sika under the name VISCOCRETE TEMPO 12 in liquid form with a color brown and a PH of 6±1, The density of this adjuvant is 1,06.

Mortars mixtures and test program

Mortars mixtures: The mortar of this study is composed by

weight, of one part cement, three parts of sand and water/cement ratio=0, 5.

Specimens and test program: The experimental study is carried out on prismatic specimens of dimensions $(4 \times 4 \times 16)$ cm. The specimens are prepared according to standard NF EN 12390-1 [25]. The approach of this comparative study consists of quantifying over time the resistance to external sulphate attack characterized by the physico-mechanical and microstructural degradation of mortars based on blast furnace slag or marble, as a substitution partial with portland cement, preserved in water and in a watermagnesium sulfate solution (MgSo₄7H₂O) with a concentration of 50g/l with renewal of the solutions and PH control.

The evolution of mechanical resistance over time is obtained by using tensile, flexural and compressive strength measurements on specimens produced according to standards NF EN 12390-5 [25] and NF EN 12390- 3 [26]. The loss of weight of the samples according to the three preservation methods is carried out on prismatic test pieces of size (4×4×16) cm. Weighing is carried out using an electronic balance. The weight loss is determined according to the following formula:

$$LW = W_1 - W_2 / W_1 \times 100\%$$

LM: Loss of weight in (%)

W₁: weight of the specimen after demoulding in (g)

W₂: weight of the test piece at different times in (g).

Result

Case of conservation in water



Figure 2: Show the effect of the nature and the rate of mineral admixtures (GGBFS or silica fume) as partial substitution to cement and the conservation mode in water.

Compressive strength: Figure 2 show the effect of the nature and the rate of mineral admixtures (GGBFS or silica fume) as partial substitution to cement and the conservation mode in water. The

rate of strength development in mortars depends on the pozzolanic and hydraulic activity of mineral admixtures.

After 60 days, a progressive evolution of mechanical strengths with curing time only on the mortars with silica fume or GGBFS. On the other hand, the reference mortar presents a stationary or slow progression. The compressive strength varied from 45.2MPa and 43.74MPa for mortars with 0.5% PET and 1% PET respectively and silica fume, to 44.71MPa and 43.23MPa for mortars with 0.5% PET and 1% PET and GGBFS. The increase in the compressive strength could be attributed firstly to the pozzolanic and hydraulic effect of mineral admixtures; the compressive strength of mortars with silica fume is higher than that with GGBFS, due to the high fineness and high silica content of silica fume compared to GGBFS, at normal temperature, the pozzolanic reaction of GGBFS is a slow process also the difference in the hydration process of the two mineral admixtures. The hydration in the presence of silica fume can be divided into two phases: the first one is characterized by a rapid hydration and dissolution accompanied by a consumption of silica fume particles and an increase of mechanical strength. The second phase is characterized by a small change of hydration but at the same time the system becomes denser as a result of the hydration products rearrangement and the change of large pores into fine pores due to the pozzolanic reaction, which has an important role

in the strength increase. In contrast, the vitrified blast furnace slag is rapidly soluble in alkaline water and therefore needs the addition of an activating agent to develop satisfactory kinetic hydration (in this case is the cement).

The progress in the compressive strength of mortars with GGBFS compared with control mortar can be explained by the finesse, specific surface area and latent hydraulic activity of GGBFS. These results are in good agreement with the literature [27]. The addition of PET fibers does not provide any appreciable improvement in compression and that the resistance is only slightly affected as shown by a certain number of studies. Other authors disagree and speak either of an increase of 15 to 20% or sometimes of a slight decrease. The fibers only play a significant role after the breakdown of the cement matrix: the energy dissipated, characterized by the surface area, is very clearly increased.

Flexural tensile strength: According to Figure 3 we can see that the compressive strength of mortars is highly influenced by the nature and the substitution rate of mineral admixtures. The type of mineral admixtures has a very slightly influence on the flexural behavior. The addition of fibers to the mortar has more marked consequences on the tensile strength. we note that, with the addition of 1% PET fibers, the flexural strength increases compared to that of the control mortar.



Figure 3: Flexural tensile strength of mortars conserved in water.

The results also show a ductile behavior of the fiber mortar. The presence of PET fibers in the matrix considerably modifies the brittle behavior by limiting the phenomenon of rapid and unstable propagation of the rupture. Their presence not only makes the propagation of the crack slower and progressive with the increase in the load but allows a transfer of stress across the lips. This allows the composite to have post-cracking resistance and to withstand much greater deformations than the matrix alone.

Case of conservation in magnesium sulphate solution

after demoulding

Loss of weight: Figure 4 show the loss of weight of mortars conserved in magnesium sulphate solution after release, we can see that all mortars show the same variations in mass. Up to 90 days, we note a significant increase in weight for all preserved study mortars directly in the sulphate solution after demoulding. This increase is obtained following the formation of brucite $Mg(OH)_2$, ettringite and secondary gypsum which results from the reaction between portlandite Ca(OH)₂ and MgSO₄.



Figure 4: Loss of weight of mortars conserved in magnesium sulphate solution after demoulding.

The increase in weight differs from mortar with GGBFS to another with silica fume. The mortars with silica fume present a slight variation in mass. This can be explained by the reduction in the quantity of aluminates tricalciums (C_3A) in the cement and consequently a reduction in the quantity of ettringite formed. After 90 days we can note a reduction in mass, this reduction is due to the leaching of portlandite Ca(OH)₂. Preserving the mortar samples in the weak sulfate solution causes the dissolution of the hydration products of the cement matrix, mainly portlandite, which causes and generates the porosity of the different mortars over time. We can see also that the PET fibers are not affected by sulfates and do not undergo any degradation.

Mechanical strength

Figures 5 & 6 shows the Flexural tensile strength and the

compressive strength of mortars conserved in magnesium sulphate solution after demoulding respectively. From Figures 5 & 6, It could be observed up to 90 days that the emergence of mortar samples in the sulphate solution causes the formation of brucite $Mg(OH)_2$ on the outer layer of the mortars which can temporarily delay the penetration of sulphate ions and therefore the evolution of the hydration kinetics. The addition of mineral admixtures improves the mechanical resistance of mortars exposed to the sulphate solution; this is essentially due to the hydraulic activity of slag and the pozzolanic activity of silica fume which generates the formation of CSH secondary and consequently the densification of the cement matrix. After 90 days, there is a reduction in mechanical resistance for all the mortars studied. Mortars with silica fume show the lowest reduction in resistance.



Figure 5: Flexural tensile strength of mortars conserved in magnesium sulphate solution after demoulding.



Figure 6: Compressive strength of mortars conserved in magnesium sulphate solution after demoulding.

The good resistance of mortars based on silica fume to external sulphate attacks is essentially due to the pozzolanic activity of silica fume characterized by consumption of part of the portlandite and formation of CSH. We can also note that our Algerian GGBFS crushed and mixed with water does not hydrate, due to the formation of an acidic layer around the slag grain preventing it from hydrating. During the hydration of the cement, the portlandite formed dissolves the acid layer around the slag grain to form secondary hydrates and reduces the PH of the mixture and consequently improves resistance in aggressive environments. exposed to the sulfate solution after 365 days of exposure. A white layer of brucite is observed on the exterior surface of the samples. The cracks and bursting observed on the mortar samples are attributed to the formation of expansive salts obtained following reactions between portlandite and magnesium sulfates MgSO₄. Magnesium sulfates are aggressive due to a double action of the Mg²⁺ cation with Ca²⁺ cations and SO₄ anions. the exchanges of Mg²⁺ with Ca²⁺ gives the formation of Mg (OH)₂ brucite which temporarily slows down the penetration of sulfates. They cause the transformation of CSH into hydrated magnesium silicate MSH.

Visual observations

Figure 7 shows the visual observations of the mortar specimens



Figure 7: Visual observation of mortars exposed to magnesium sulphate solution.

SEM observations

Microstructural analyzes were carried out on samples of reference mortar, mortar with GGBFS and those with silica fume preserved for one year in a solution of magnesium sulfate MgSO₄. This analysis allowed us to identify the new products obtained following reactions between sulfate ions and cement hydration products. Figure 8A shows that the microstructure of the control

mortar preserved in the $MgSO_4$ solution is very affected by the sulphate attack, which results in the formation of ettringite crystals in balls. We can see in Figure 8B the presence of brucite on the surface of the sample, the latter was formed following the reaction of portlandite CH and the $MgSO_4$ solution. This justified the absence of portlandite on the sample surface. Figure 8C shows the presence of the CSH gel.



Figure 8: SEM observation of mortars exposed to magnesium sulphate solution.

Conclusion

The study presented in this paper is a contribution to a better understanding on the effects of mineral admixtures and PET fibers on the durability of mortars. PET fibers and silica fume or GGBFS were investigated. The following conclusions are drawn based on the results of different tests and analysis:

- a. The mechanical resistance of mortars preserved in MgSO₄ solution is very influenced by the nature of the mineral admixtures. For the period up to 90 days, the mechanical resistances are slightly affected by external sulphate attacks. This is explained by the formation of an exterior layer of brucite, which serves to delay the penetration of sulfate ions and consequently the continuity of the hydration process.
- b. Beyond 90 days, a drop in mechanical resistance and visible degradation was noted. This is mainly due to the formation of gypsum and secondary ettringite which causes the expansion and consequently a low resistance and the appearance of cracks on the outer layer of the mortar specimens.
- c. The PET fibers are not affected by sulfates and do not undergo any degradation.

- d. The effect of substitution of silica fume and GGBFS improves the mechanical resistance of mortars to sulphate attack. The mortars with silica fume present the lowest reductions in mechanical resistance as well as that with GGBFS compared to the control mortar. This improvement is mainly due to the pozzolanic and hydraulic activity of silica fume and GGBFS which serves by the consumption of part of the portlandite and the formation of HSC.
- e. The microstructural analysis of control mortar samples and with mineral additions makes it possible to identify the reaction products between sulfate ions and cement hydration products to justify the results obtained. By analysis of the SEM photos we note that after one year of conservation of the mortars in a sulphate medium the test pieces have suffered superficial degradation due to the formation of expansive products.

Acknowledgment

The authors extend their appreciation to the Higher Normal School of Technological Teaching of Skikda, Algeria and the Laboratory of Anticorrosion-Materials, Environmental and Structure "LAMES", University of 20 August 1955 Skikda, Algeria, and the Materials, Geomaterials and Environment Laboratory (LMGE), Department of Civil Engineering, Badji Mokhtar- Annaba Algeria for the use of their lab facilities.

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