Durability assessments of limestone mortars containing polypropylene fibres waste

Khadra Bendjillali¹, Bensaid Boulekbache² and Mohamed Chemrouk³

¹Department of Civil Engineering, Laboratory of Structures Rehabilitation and Materials, University Amar Telidji, P.O. Box 37G, Route de Ghardaia, Laghouat 03000, Algeria
²Department of Civil Engineering, Laboratory of Materials Sciences and Environment, University Hissabia Benbouali, Chief, Algeria
³Department of Structure and Materials, Laboratory of Buildings in the Environment, University of Sciences and Technology Houari Boumediene, Algiers, Algeria

(Received May 14, 2020, Revised August 5, 2020, Accepted August 6, 2020)

Abstract. The main objective of this study is the assessment of the ability of limestone mortars to resist to different chemical attacks. The ability of polypropylene (PP) fibres waste used as reinforcement of these concrete materials to enhance their durability is also studied. Crushed sand 0/2 mm which is a fine limestone residue obtained by the crushing of natural rocks in aggregates industry is used for the fabrication of the mortar. The fibres used, which are obtained from the waste of domestic plastic sweeps’ fabrication, have a length of 20 mm and a diameter ranging between 0.38 and 0.51 mm. Two weight fibres contents are used, 0.5 and 1%. The durability tests carried out in this investigation included the water absorption by capillarity, the mass variation, the flexural and the compressive strengths of the mortar specimen immersed for 366 days in 5% sodium chloride, 5% magnesium sulphate and 5% sulphuric acid solutions. A mineralogical analysis by X-ray diffraction (XRD) and a visual inspection are used for a better examination of the quality of tested mortars and for better interpretation of their behaviour in different solutions. The results indicate that the reinforcement of limestone mortar by PP fibres waste is an excellent solution to improve its chemical resistance and durability. Moreover, the presence of PP fibres waste does not affect significantly the water absorption by capillarity of mortar neither its mass variation, when exposed to chloride and sulphate solutions. While in sulphuric acid, the mass loss is higher with the presence of PP fibres waste, especially after an exposure of 180 days. The results reveal that these fibres have a considerable effect on the flexural and the compressive behaviour of mortar especially in acid solution, where a reduction of strength loss is observed. The mineralogical analysis confirms the good behaviour of mortar immersed in sulphate and chloride solutions; and shows that more gypsum is formed in mortar exposed to acid environment causing its rapid degradation. The visual observation reveals that only samples exposed to acid attack during 366 days have showed a surface damage extending over a depth of approximately 300 μm.

Keywords: fibre concrete; recycled polypropylene fibres; crushed sand; chemical attacks; capillarity; absorption, mechanical strength; expansive gel

1. Introduction

The strength, the serviceability and the aesthetic of structures are conditioned by the durability of used materials, such as concrete and mortar. The durability of these materials is defined as their ability to resist any destructive action, which could be climatic, chemical or mechanical and retain their quality and original form, when exposed to aggressive environment. Generally, concrete durability can be achieved by an adequate selection of the mixing ingredients, a good composition, a controlled placing and curing and also by a proper quality assurance of construction process as reported by Gjørv (2013). Actually the comprehension of the behaviour of cement materials facilitates the determination of the appropriate composition for each environment. In this sense, codes and standards are practical guides for construction designers.

Under certain environment conditions, concrete is threatened by salts, sulphates and acids coming from rainwater, industrial water and ground water or from foundations soil. These chemical agents can penetrate into concrete through capillary absorption or diffusion and are stored in the micro-cracks and the pore spaces of the cement paste or in the paste-aggregate interfaces and then disturb its chemical stability and its durability performance.

In marine environment, the durability of concrete depends mainly on its ability to resist to chloride ingress (Costa and Appleton 1999). The diffusivity of chloride agent into concrete which is a complex phenomenon involving numerous mechanisms, leads to rapid deterioration of reinforced concrete structures and greatly affects its service life (Wang et al. 2016). The diffusivity and the penetration of chloride are not only a function of the concrete pore structure and the presence of cracks, but also of chloride exposure conditions and the mix design parameters, such as the water/cement ratio, the type of aggregates and cement, the presence of mineral additions and the adequacy of curing. When chloride ions enter in the cement pores, they first react with portlandite Ca(OH)₂,
reducing the material alkalinity and subsequently leading to the corrosion of steel reinforcement (Koleva et al. 2007). The corrosion of steel bars that leads to the cracking and spalling of concrete cover is the main cause of structural deterioration (Ayinde et al. 2019). It is reported that the mechanical strength of concrete mixed and cured in seawater is higher than that mixed and cured in fresh water at early age (up to 14 days), while at later ages (up to 90 days), the crystallisation of salt leads to a reduction of this strength (Wegian 2010). The sodium chloride NaCl reacts with the the portlandite Ca(OH)\textsubscript{2} and with the tricalcium aluminate (C\textsc{3}A) forming (C\textsc{3}A.CaCl\textsubscript{2}.10H\textsubscript{2}O), known as Friedel’s salts. The formation of Friedel’s salts is the main cause of damage of cementitious materials exposed to NaCl. However, the relationship between the volume change due to these salts and the reduction of mechanical strength is not yet known (Qiao et al. 2018). According to the study of Peterson (1995), the sodium chloride solutions have a little effect on the compressive and flexural strengths of mortar. In the same sense, other researchers (Darwin et al. 2008) have revealed that, at lower concentration (3%), sodium chloride has a small negative effect on the concrete’s properties.

Sulphates are chemicals that may attack concrete and induce sever damages to concrete structure (Zhang et al. 2013) and its deterioration is the dominant source of external chemical attacks (Chemrouk 2015). The magnesium sulphate (MgSO\textsubscript{4}) is considered as the most harmful to cement material; it reacts with all the hydrated cement compounds. The result of this reaction is the formation of gypsum, brucite (De Schutter 2012) and ettringite. Santhanam et al. (2002) reported that the deterioration of mortar by magnesium sulphate attack is due to the decalcification of the calcium silicate hydrate (C-S-H), and its conversion to magnesium silicate hydrate (M-S-H) through the substitution of the calcium ions Ca\textsuperscript{2+} by the magnesium ions Mg\textsuperscript{2+}. The new formations M-S-H are responsible for the expansion, the cracking and then the deterioration of concrete (Hekal et al. 2002); the absence of the binding character of M-S-H causes the loss of mechanical strength. The magnesium hydroxide (brucite) Mg(OH)\textsubscript{2} is characterised by a low solubility; it precipitates into surface pores of the concrete by forming a protective layer slowing down the sulphate penetration and preventing the continuity of the reaction (Roziere 2007). The solubility of brucite is 0.01 g/l, compared to 1.37 g/l for calcium hydroxide (Prasad et al. 2006). The penetration of the sulphate solution into the mortar is function of the rate of the solution diffusion across the brucite layer (Santhanam et al. 2003).

Since concrete is an alkaline material, it reacts with different acids. Depending on the nature and concentration of acid, concrete can be rapidly or slowly disintegrated (Ashish et al. 2016). The sulphuric acid H\textsubscript{2}SO\textsubscript{4} is considered as the most aggressive acid for concrete. The high solubility of calcium salts CaSO\textsubscript{4} produced by the reaction of calcium hydroxide Ca(OH)\textsubscript{2} with the H\textsubscript{2}SO\textsubscript{4} is the main cause of concrete degradation and loss of its performance.

For enhancing the durability of concrete, it is suggested to protect its dry surface by hydrophobic materials, such as fluoropolymer, silicate resin and sodium acetate crystallising material (Al-Kheetan et al. 2019, 2020). As reported by the authors, these three materials have a good hydrophobic effect on concrete, but their efficiency in reducing water absorption is different. The introduction of an anhydrous sodium acetate material in the fresh mix is also an efficiency protection of concrete that helps to form a denser concrete structure, having minimum cracks and higher compressive strength (Al-Kheetan and Rahman 2019). The adoption of fibres can also be considered as a good solution to enhance the durability of concrete and to restrain its degradation, when it is subjected to some deterioration mechanisms. Numerous studies (Mohammadhosseini and Tahir 2018, Ramezani and Al 2013) confirm the efficiency of synthetic fibres in the reduction of the capillary absorption of concretes. Bolat et al. (2014) justified this efficiency by the capacity of synthetic fibres to gather on the surface of the material thanks to their low weight, compared to other type of fibres and then prevent the penetration of aggressive elements. Synthetic fibres of polypropylene (PP) nowadays are the most used fibres in different technical fields (Nuaklong et al. 2020), because they possess considerable mechanical characteristics and an excellent chemical stability (Stakne et al. 2003); they also have a low cost and a high shrinkage cracking resistance. Thanks to their high resistance to corrosion, PP fibres have a long-term durability (Yin et al. 2015). By forming a network to restrict the growth of Ca(OH)\textsubscript{2}; crystalline and reduce micro-voids, PP fibres condense the microstructure of concrete (Sun and Xu 2009). They significantly improve the flexural and splitting tensile strengths of concrete and also its impermeability (Zhang and Zhao 2012). Due to their ability to restrain the extension of cracks, reduce the extent of stress concentration at the tip of cracks and delay the growth rate of cracks, PP fibres reduce the water absorption of concrete and enhance its mechanical properties (Afrighsabet and Ozbakkalaglu 2015); they also delay the diffusion of sulphate and chloride ions into the matrix (Nguyen et al. 2019). According to some authors (Nili and Afrighsabet 2012), the reduction of the material’s water absorption by the use of PP fibres is due to their capacity to limit both the number and the width of cracks. The reinforcement of concrete stored in a chloride environment with PP fibres delays its degradation by reducing its permeability (Afrighsabet et al. 2016), its shrinkage and its expansion (Kakoei et al. 2012).

Today, with the increase of the concrete consumption, it becomes necessary to find alternative materials that can be used for its production (Faraj et al. 2020, Paliwal and Maru 2017) and for its reinforcement (Al-Hadithi and Abbas 2018). The recycling of wastes not only constitutes new resources of these materials, but also participates to protect the public health and reduce the environment pollution.

The present study which is in line with this axis of research is a contribution for proposing an environmentally friendly and a sustainable construction material. This new material is a composite fabricated by a matrix based on natural residue, used as fine sand and a reinforcement based on synthetic fibre waste. Specifically, this study contributes to find efficient solutions for resolving some environmental
problems caused by the presence of plastic wastes in nature or the loss of the ecological equilibrium through the disappearance of certain natural resources. The study also contributes to resolving some technical problems commonly occurring in concrete structures such as cracking and durability of the cementitious materials.

River sand is the most used sand in the concrete fabrication and road construction, despite the existence of another fine sandy material in large quantities without any exploitation. This material is a fine residue (0/2 mm) obtained by the crushing of limestone rocks for the fabrication of aggregates 3/8, 8/15 and 15/25 mm. In Algeria, the gravel industry generates about 15 million tons of crushed limestone sand as a by-product (Meziane et al. 2015). Generally, this crushed sand is not used in construction because it contains a high percentage of fines. This fine’s residue is disposed off in the open environment without any ecological study on the impact of such disposal, hence causing serious environmental problems. The present study is an attempt to evaluate the performance of mortar prepared with this residue. The use of limestone sand can be a good choice in this work because it participates in different chemical reactions in the same way as cement, since they contain the same main component, which is the calcite. This positive participation can reduce significantly the severity of chemical attacks. Skaropoulou et al. (2012) have already noticed that the use of limestone aggregates in concrete improves its sulphate resistance.

Due to the arid climate and aggressive environment in certain regions, the concrete suffers from shrinkage problems caused by the excessive evaporation of mixing water, which leads to the apparition of cracks on the surface of the structural elements. These cracks become an easy way for the penetration of different aggressive solutions, the consequence of which is rapid degradation of structures. According to all literature references, the addition of fibres into fresh mortar is an excellent solution to control shrinkage cracks and then enhance the durability of the material. Since it is a non-biodegradable material, releasing harmful gases when burned (Karanth et al. 2017), the recycling of plastic fibres coming from industrial waste can offer new resources for durable and economical construction materials and an efficient environmental protection.

This experimental research presents the results of the study of the ability of limestone mortar reinforced by PP fibres, coming from plastic sweeps’ fabrication waste to resist to chloride (NaCl), sulphate (MgSO₄) and acid (H₂SO₄) attacks. Two weight dosages of fibres are used, 0.5 and 1% of the total mortar’s mass. The water capillarity, the mass variation, the flexural strength and the compressive strength are measured on mortar samples from 14 days to 366 days of exposition to chemical solutions; mineralogical analysis by X-ray diffraction (XRD) and visual examinations were carried out on the tested specimens.

2. Experimental procedure

2.1 Materials

The materials used for the tested mortars consisted of Portland cement CEM II/A 42.5, having specific surface of 3200 cm²/g and density of 3 g/cm³, crushed limestone sand 0/2 mm having the properties shown in Table 1 and mineralogical composition illustrated in Fig. 1 PP fibres as shown in Fig. 2, having the properties summarised in Table 2. The chemical analysis of sand revealed the presence of 90% of CaCO₃, 0.51% of SO₄²⁻ and 6.8% of insoluble materials. The fibres used in this investigation are synthetic fibres of polypropylene obtained from the waste of the fabrication of domestic plastic sweeps.

Control mortars are prepared according to EN 196-1 (2005), with a sand/cement ratio of 3 and a water/cement ratio of 0.55. Generally, using fibres in cementitious mixtures leads to loss of their workability and the solution in this case is the addition of an appropriate amount of superplasticizer, as recommended by Söylev and Özturan (2014). The dosage of the superplasticizer that gives workable mortars is about 2.88% by weight of cement. Two weight contents of PP fibres waste are tested in this work, 0.5 and 1 wt.%.

### Table 1 Physical properties of limestone sand

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent density</td>
<td>1.45 g/cm³</td>
</tr>
<tr>
<td>Specific density</td>
<td>2.52 g/cm³</td>
</tr>
<tr>
<td>Compactness</td>
<td>58 (%)</td>
</tr>
<tr>
<td>Absorption coefficient</td>
<td>4.50 (%)</td>
</tr>
<tr>
<td>Sand equivalent</td>
<td>63 (%)</td>
</tr>
<tr>
<td>Methylene blue value</td>
<td>0.13 ml/g</td>
</tr>
<tr>
<td>Fineness modulus</td>
<td>1.80</td>
</tr>
</tbody>
</table>

### Table 2 The chemical analysis of sand revealed the presence of

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaCO₃</td>
<td>90%</td>
</tr>
<tr>
<td>SO₄²⁻</td>
<td>0.51%</td>
</tr>
<tr>
<td>Insoluble</td>
<td>6.8%</td>
</tr>
</tbody>
</table>

### Fig. 1 X-ray diffraction of limestone sand

### Fig. 2 PP fibres waste used
2.2 Samples and testing programs

After the moulding of the mortar prisms with size of 40×40×160 mm, the moulds are covered with a plastic film and the specimens were demoulded 24 hours later. Thereafter, all prisms are cured in saturated limewater at 20±2°C, until they reached a compressive strength of 20MPa as described by the Standard ASTM C 1012 (2004). After this initial curing, mortar specimens are stored at 20±2°C in 5% sodium chloride solution (NaCl), in 5% magnesium sulphate solution (MgSO₄), in 5% sulphuric acid solution (H₂SO₄) and in potable water until testing. The conservation tanks must be covered to prevent evaporation and the solutions must be renewed every 30 days.

A total of 231 prisms with dimensions of 40×40×160 mm were cast: 6 were tested for capillary water absorption after a period of 0, 2, 4, 6, 8, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90 and 180 min (three hours) and 225 for flexural strength, after an immersion time of 0, 14, 28, 56, 91, 180 and 366 days (the results of 03 samples for calculating the flexural strength). The compression test was carried out on the resulting half prisms of the flexural test (the results of 06 samples for calculating the compressive strength).

It is very important to note that to study the durability performance of concrete, it is necessary to measure the capillary absorption, because it constitutes the most efficient way to transport chloride and sulphate ions Wittmann and Zhao (2012). The water absorption by capillarity is due to the difference between the fluid’s surface capillary pressure and its gravity pressure, which induces fluid movement until a balance is established. The test is realised according to EN 13057 (2002) on dry samples of 28 days aging. The capillary water absorption (i) is calculated between the periods of 2 to 180 minutes from the starting of the test by using the following expression

\[ i = \frac{W}{A} \]  

(1)

where \( i \) is the capillary water absorption (g/mm²), \( W \) is the amount of water adsorbed (g) and \( A \) is the cross-section of specimen that was in contact with water (40×40 mm²).

Mortar samples were weighed with a precision balance 0.01 g after 7, 14, 21, 28, 56, 91, 180, 270 and 366 days of chemical exposure. The mass variation is determined by the following relation

\[ \% \text{mass variation} = \frac{m - m_0}{m_0} \times 100 \]  

(2)

where \( m_0 \) is the weight of the sample before immersion and \( m \) is the weight of the sample after immersion.

Three points bending and compression tests are conducted after 14, 28, 56, 91, 180 and 366 days of chemical immersion. X-ray diffraction (XRD) analysis is used to find the products formed by hydration and chemical reactions in mortar immersed in different solutions during one year. Weekly visual observation is performed on the samples to evaluate the degradation of mortars.

3. Experimental results

3.1 Capillary water absorption

Fig. 3 shows the capillary water absorption of mortars. All mortars with and without PP fibres waste have presented almost the same capillary absorption, which is around 2.7×10⁻³ g/mm² over the first three hours. Generally, concretes with less capillarity pores are characterised by better performances (Zhang et al. 2020). The low capillarity which indicated good quality of materials can be attributed to the reduced porosity in mortar’s mass, because under good conservation conditions; the concrete reaches its most compact structure at 28 days.

The development of C-S-H gel in hardened concrete reduces the porosity, as a result of which the water capillary decreases and hence the durability is improved (Ramadoss and Nagamani 2008). The reduced porosity is not only due to the ability of used PP fibres waste to occupy pores and to improve its compactness thanks to their flexibility, but also to the presence of calcareous fines in the crushed sand, which reduce the material porosity by ensuring the maximum compactness and hence improve its impermeability by blocking the passages connecting capillarity pores, as reported by Meziane et al. (2015). Pereira de Oliveira and Castro-Gomes (2011) confirmed through their study that the introduction of fibres into mortar mass modifies its porosity structure, especially in fibres-matrix zone and then reducing the number of capillarity pores.

3.2 Mass variation

The variation in the mass of the material exposed to aggressive environment is a good durability indicator. The

Table 2 Properties of PP fibres waste

<table>
<thead>
<tr>
<th>Surface texture</th>
<th>Smooth</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cross-section shape</td>
<td>Circular</td>
</tr>
<tr>
<td>Chemical resistance level</td>
<td>High</td>
</tr>
<tr>
<td>Average length</td>
<td>20 ± 2 (mm)</td>
</tr>
<tr>
<td>Average diameter</td>
<td>0.38 – 0.51 (mm)</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>0.99 (g/cm³)</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>210 - 250 (MPa)</td>
</tr>
<tr>
<td>Elasticity modulus</td>
<td>4 - 5 (GPa)</td>
</tr>
<tr>
<td>Fusion temperature</td>
<td>300 (°C)</td>
</tr>
</tbody>
</table>

Fig. 3 Capillary water absorption of mortars
more the material dissolution is important, the more the mass loss is high and the deterioration is rapid. Figs. 4-6 show the variation of the mass in control mortar, in 0.5% PP fibres waste mortar and in 1% PP fibres waste mortar, respectively, immersed in different chemical solutions. It is observed that all mortars with and without PP fibres waste immersed in potable water and in sodium chloride and magnesium sulphate solutions have gained a mass; while mortar samples exposed to sulphuric acid solution have showed a high loss of their mass from the 180th day of exposure. The increase of weight observed especially in water and in sodium chloride and magnesium sulphate solutions is due to the products formed during cement hydration, essentially an ettringite and C-S-H gel. In the same sense, Massaad et al. (2016) suggest that the variation of mass during a sulphate attack is due to the leaching and precipitation of minerals, the aggregates loss and the infiltration of water in the cracks. From Figs. 4-6, it can be seen that the mass variation of mortars is very close in potable water and in sodium chloride solution; it does not exceed 1.6%, contrary to that in magnesium sulphate solution, when it is more than 2.16% after 366 days of exposure. The gain of mass in mortars stored in 5% of MgSO_4 is previously confirmed by other researchers (Aziez and Bezzar 2017, Kilincakle 1997). As confirmed in recent study (Zhang et al. 2020), the mass variation of concrete conserved in sulphate environment depends of the evolution of mesoscopic pore structures. Once again, it is shown in an experimental study conducted by Yildirim and Sumer (2013) that the absorption of cement mortars immersed in MgSO_4 solution is higher than that measured in NaCl solution. Whereas other authors (Roziere et al. 2009) have noted a lower increase or some time slight loss in the mass of concrete exposed to sulphate.

It is very important to note that the effect of PP fibres waste is observed much more in acid solution; when the mortar without PP fibres waste reaches the highest mass gain which is in the order of 4%, after 180 days and a loss of 6% of its weight, after one year of immersion, whereas with presence of PP fibres waste, the mass loss exceeds 8.7% after 366 days of immersion in acid solution. As explained by Madhuri and Srinivasarao Rao (2018), this greater mass loss is due to the formation of gypsum resulting from the reaction between sulphuric acid and calcium hydroxide of concrete. This high loss of mass confirms once again the severity of sulphuric acid, which is a source of two attacks, one acidic and other sulphatic and also confirms the adverse effect of the high concentrations of acid solution. The concentration of 5%, used in this investigation, seems to be very damaging for mortars, since it favours the formation of more expansive products which are responsible for the increase of volume and then the decrease of material density. So the presence of mortar in sulphuric acid solution, which is considered as the most aggressive acid for cimentitious materials leads to the formation of expansive materials such as gypsum (calcium salts). These salts are characterised by a high solubility that accelerates the mortar degradation. Firstly the mortar of the surface decomposes, and as a result, fibres are released from the matrix that surrounds them. So in these conditions, the penetration of the acid solution is very easy through these new channels formed in the decomposed matrix; the released PP fibres with smooth surfaces would help the circulation of the acid solution, which explain the increase...
of loss of mass in the PP fibres waste mortar.

### 3.3 Flexural strength

Figs. 7-9 show the results of flexural strength for mortar specimens exposed to different chemical solutions until 366 days. In both water and sodium chloride, mortars continued gaining flexural strength; but in sulphuric acid and magnesium sulphate solutions, a strength decrease is observed beyond 14 and 28 days of exposure, respectively. The strengths of mortars exposed to sulphate and acid solutions during one year are usually higher than reference values, reached before any exposure, with a percentage of 43% and more than 50% in control and PP fibres waste mortar, respectively. The strengths of mortars exposed to sulphate and acid solutions during one year are usually higher than reference values, reached before any exposure, with a percentage of 43% and more than 50% in control and PP fibres waste mortar, respectively. These percentages are calculated after one year exposure with regard to the maximal values reached at the 14th day exposure. As seen in Fig. 10(a), the addition of 0.5% PP fibres waste improves the flexural resistance of mortars by 10, 2, 14 and 42% after one year immersion in potable water, NaCl, MgSO4 and H2SO4 solution, respectively. With 1% PP fibres waste, the improvement becomes 19, 11, 27 and 65%, respectively, according to Fig. 10(b). The resistance of the transition zone matrix-fibre is also positively affected by C-S-H resulting from hydration reaction, which explains the good behaviour of different mortars towards tensile actions. The positive role of this type of fibrous waste on the mechanical behaviour of mortar and concrete has been previously confirmed (Bendjillali and Chemrouk 2018, Bendjillali et al. 2019).

The good adhesion between the matrix and the PP fibres waste participates to the improvement of the flexural strength of the mortar, since an insufficient bonding can lead to an easy fibre pull out failures, as noted by Ghaffar et al. (2020). The PP fibres used in this work have a smooth surface; from a theoretical point of view, they do not assure a good adherence and a sufficient mechanical anchorage, by
comparison to curled fibres. However, due to the relatively better workability obtained in mixes with smooth fibres by comparison to that obtained in mixes with curled fibres, and thanks to the implementation of mortar with smooth fibres which is more easy and adequate, the dispersion of this type of smooth surface fibres in the mortar mix is more homogeneous and uniform than that with curled fibres. This makes the mechanical gain obtained with smooth surface fibres relatively more important and flexural strengths become more appreciable. This conclusion which is in accordance with the review presented by Soroushian and Bayasi (1991) is further asserted in another experimental work carried out by the author (Bendjillali 2015) on the mechanical behaviour of mortar reinforced by two types of PP fibres, smooth straight fibres and curled fibres. The results obtained have shown that the flexural behaviour is improved more with smooth straight fibres. The improvement was between 25 and 8% with a reinforcement of smooth straight and curled respectively (At 90 days, for 1% of PP fibres), compared to mortar without PP fibres. Close observations of the tested specimens showed the absence of any fibre pull out during all the flexural tests carried out in this work. This revealed that the interfacial bonding between the used fibres and the cement paste has reached a desirable level to result in a better flexural strength and toughness; probably this was also helped by the sufficient anchorage length of the fibres without negatively affecting the workability of the mortar mix. Pakravan et al. (2012) have reported that the mechanical interactions of PP fibres with matrix are the most important in the adhesion energy. According to the study of Kalinowski and Trägårdh (2007), the dense interfacial transition zone in PP fibre concrete is formed by the portlandite nucleation which is favours by the surface of PP fibres. It is confirmed by Singh et al. (2004) that the increase of the interface fibre-matrix resistance in salt exposure is due to the increase of friction forces between fibres and matrix. In the same sense, the used PP fibres waste do not participate in any chemical reaction due to their high chemical resistance; nevertheless they prevent efficiently the entrance of chemical agents into the material by their anti-cracking effect, as reported by Zhang and Li (2013) and then help to maintain its mechanical performance.

On other hand, according to Fig. 3, the low capillarity that does not exceed 0.01g/mm² in both PP fibres waste mortar and control mortar explains the capacity of these materials for resisting to the penetration of the chemical solutions tested in this work, and then for maintaining their mechanical resistance even after one year of exposure.

3.4 Compressive strength

From Figs. 11-13, compressive strength of different mortars increases gradually with time, in both potable water and in NaCl solutions. On the contrary, the specimens exposed to MgSO₄ and H₂SO₄ solutions have exhibited a reduction of their compressive strength after an exposure of 28 days. At 366 days in sulphate solution, this reduction is about 11% and 8.5% in control mortar and 1% PP fibres waste mortar, respectively, with regard to maximal compressive strengths obtained after 28 days exposure; while in acid solution, the reduction drops from 23% in control mortar to 17% in 1% PP fibres waste mortar. Lee et al. (2008) have reported that in tap water, the compressive strength tends to stabilise, however in magnesium sulphate solution it decreases after 91 days of exposure. The decrease of the compressive strength of mortar in MgSO₄ and H₂SO₄ solutions is less important than that of its flexural strength; this may be due to the higher sensitivity of tensile strength to cracking, compared with compressive strength, as explained by Boyd and Mindess (2004).

The reduction of the mechanical strength under the effect of magnesium and acid solutions is probably due to the decomposition of cement hydrates, such as C-S-H and portlandite. The rapid dissolution of the portlandite, which is the first hydrate product to dissolve, has a little effect on the mechanical strength of material, but it increases its
porosity and then facilitates the penetration of aggressive ions. The immersion of concrete in water is considered as an ideal curing for obtaining the best compressive behaviour (Bendjillali and Makhloufi 2012). By comparison with reference strengths values reached before any exposure, the increase percentage after one year reached 20% in control mortar and between 26 and 28% in both 0.5 and 1% PP fibres waste mortar, respectively in potable water and between 12, 16 and 15%, respectively in NaCl solution. The continuous increase of the compressive strength in these two solutions is certainly due to the increase of the rate of hydration, which participates to develop structure by reducing the porosity and to gain strength and then limit the destructive effects of aggressive attacks.

Contrary to flexural strengths, compressive strengths obtained after 366 days exposure in sulphate and acid solutions are less than reference values, with a percentage reduction between 7% in mortar without fibres and only 3% in 1% fibres waste mortar for sulphate solution and between 19% and 14% in mortar without fibres and 1% fibres waste mortar respectively for acid solution. The high strength reduction of mortars under acid attack can be related to weight loss which reaches 8.7% in PP fibres waste mortar.

The compressive strength of mortars can be related to their capillarity, which constitutes a good indicator of capillarity pores volume; the more the capillarity of mortar is lower, the more its compressive strength is higher. All these results confirm the beneficial effect of PP fibres waste primary to improve the mechanical strengths of mortar and secondly to assure its durability when it is exposed to aggressive solutions. According to Fig. 14(a), when mortar is reinforced by 0.5% PP fibres waste, the increase of its compressive strength does not reach 9% after one year of exposure in different solutions, by comparison to control mortar. While with a reinforcement of 1% PP fibres waste, the average increase is 12% in all tested solutions, as shown in Fig. 14(b). This better behaviour is due to the ability of PP fibres waste to fill free spaces and also to the positive effect of limestone fines presented in crushed sand, which ensure a good cohesion with the cement paste and a high compactness of mortars. Berredjem et al. (2020) have confirmed that the continuous evolution of compressive strength is due to the good quality of the microstructure of the interfacial transition zone of limestone aggregates which is improved by chemical reactions occurring with the cement. It is previously confirmed that the use of limestone aggregates for the fabrication of concrete improves considerably its sulphate resistance (Skaropoulou et al. 2012). The beneficial effect of limestone aggregates in sulphuric acid solutions was also confirmed by Makhloufi et al. (2012), where the authors have concluded that the acid solution was partially neutralized by the cement alkalis and the limestone aggregates.
3.5 XRD analysis

The diffractograms obtained by X-ray diffraction of mortars’ surface conserved during one year in different solutions by comparison to their reference state are shown in Fig. 15. The diffractogram of reference is that of mortars conserved in limewater before any exposure. The largest peak which appears in all the diffractograms is that of the calcite coming from limestone sand used in the fabrication of tested mortars. The portlandite amount is lower in mortars conserved in different solutions, by comparison with the mortars of reference, which can explain its use for the formation of gypsum and calcium sulphate. This result is very clear since that the portlandite is the first hydrate to dissociate once the pH of environment is less of that of concrete.

As seen in Fig. 15(a), the majority of peaks are occupied by the gypsum, because the exposition to H₂SO₄ constitutes a combination of two attacks, one acidic and other sulphatic, which favour the formation of more gypsum. The result is in agreement with that reported by Liu et al. (2015), wherever the authors have justified the high amount of gypsum by the effect of acidic solution pH. In addition new peaks of gypsum, ettringite and C-S-H appeared in acid diffractogram by comparing to reference diffractogram. It should be noted that the reaction between the acid and the calcium coming from limestone sand provides a new source of gypsum. As reported by Lakhssassi et al. (2019), the formation of certain expansive materials as ettringite and gypsum is the main cause of the decomposition of Portland cements in sulphuric acid that leads to the cracking and bursting of cement paste.

According to De Schutter (2012), the degradation of cementitious materials in magnesium sulphate is due to the formation of brucite Mg(OH)₂ which reduces the pH of the interstitial solution and then accelerates the dissolution of the portlandite that expands, causing the cracking of the material (de Larrard 2010). By observing the diffractogram of Fig. 15(b), only traces of brucite are detected, which justifies the good resistance of mortars to sulphate attacks and confirms the results of their mechanical behaviour. Generally, in the case of a sulphate attack, there is a transfer of sulphate ions to the cement matrix and a leaching of calcium ions to the external solution, as reported by Ragoug et al. (2019). It seems that thanks to the limestone nature of used sand which is composed essentially by calcium carbonate, the transfer phenomenon is very slow and then no deterioration is observed on the mortar. News peaks of calcium sulphate appear on the diffractotogram corresponding to sulphate solution, which explains the nature of the fine layer formed on the samples’ surface. It is about non hydrated gypsum that constitutes a protective layer preventing the sulphate penetration.

From the diffractogram of Fig. 15(c), only some traces of calcium chloride resulting from the reaction between the portlandite and the sodium chloride are detected. This observation justified the non presence of Friedel’s salt, which is considered the first responsible of deterioration of material in sodium chloride. The apparition of new peaks of C-S-H in this diffractogram can explain once again the
good resistance of mortar in chloride solution.

Some new peaks of C-S-H are observed in the diffractogram of Fig. 15(d), which justified the continuous evolution of the mechanical strength of mortars conserved in potable water.

3.6 Visual inspection

A visual inspection is made on mortar samples for evaluating their deterioration degree under the effect of chemical attacks. No damage such as cracking, scaling and expansion is observed on the mortar samples immersed during 366 days in chloride or sulphate solutions. Comparable results have been obtained in previous study (Aziez and Bezzar 2017), where the authors didn’t observe any damage on mortar exposed to sulphate attack for 12 months. As can be seen from Fig. 16(a), only samples immersed in acid solution have showed a damage of their surface extending over a depth of about 300 µm. A surface scaling is observed on all mortars faces. A dense white deposit covers the sample surface and a high quantity of this deposit is decanted in the conservation tank. As shown in Fig. 16(b), the X-ray diffraction revealed that it is about gypsum, i.e., calcium hydrate sulphate. This salt which is produced by the reaction of sulphuric acid and calcium hydroxide Ca(OH)_{2} coming from cement is considered as the main cause of degradation of cementitious materials exposed to acidic attack. It is well known that the more the pH of environment is low, the more the concrete damage is important. The pH of acid solution before the immersion of mortar samples was almost 1.5; this value is lower than that of the matrix (pH=13). The monthly renewal of this solution ensures the continuity of ions transfer from interstitial solution to external environment and thus the continuity of degradation reaction. No other deterioration sign such as cracking is detected below damaged surface of mortars immersed in H_{2}SO_{4}; it is probably due to the capacity of gypsum layer formed on the mortar’s surface to slow down this chemical attack, as reported in the literature. It should be noted that the sand of limestone nature and the cement used in the mortar fabrication provide a good solution to the protection of the environment.

As can be seen from Fig. 16(c), the only changes observed on the surface of samples exposed to MgSO_{4} are the formation of a white deposit, without any deterioration sign. It is probably about a protective film formed by non-hydrated gypsum that tends to waterproof the surface of mortars; as reported by Neville (2004) and stops the chemical reaction by slowing the penetration of sulphate.

By observing the surface of mortars exposed to NaCl, as shown in Fig. 16(d), the only signs observed are the formation of dark brown stains with the deposit of a very fine white precipitate on the walls of the conservation tank.

The samples of mortar conserved in potable water have saved their original colour and surface appearance, without any change or damage. According to de Larrard (2010), the dissolution of cement hydrates in water is a very slow process that takes hundreds of years.

PP fibres waste incorporated in the mortar have not deteriorated by the used aggressive solutions, which approves once again the appreciated resistance of this type of fibres to different chemical environments and also to alkaline environment of concrete.

Based on these visual observations, there is an excellent correspondence between the mechanical characteristics and the visual damage observed on the surface of the mortar samples. In sulphate solution, mortars’ strength is not significantly reduced after one year of attack and their surface have not presented any damage; while in acid solution, the visual appearance of the mortar samples is in itself an evidence of the reduction of their strength.

According to the review presented by Yi et al. (2020), the depth of chemical solution penetration, such as chloride and sulphate depends to the multiple interactions with concrete components. The findings of this study are in accordance with this review, since the used components have participated successfully for enhancing the durability of the tested material.

4. Conclusions

Based on the experimental results obtained in this investigation, the following conclusions can be made:

- The valorisation of fine limestone residue in the fabrication of mortar is an excellent solution to prevent the loss of a natural material in front of high need of aggregate in the construction sector and to participate to the protection of the environment.
- The recycling of PP fibres waste obtained from domestic plastic sweeps’ fabrication for the reinforcement of mortar provides a good solution to manage this domestic waste, to reduce the mortar’s fabrication cost and to improve its chemical resistance and durability.
- All mortars with and without PP fibres waste have presented almost the same capillary absorption, which means that this waste have no significant effect on this property.
- A gain of mass is recorded in all mortars immersed in potable water, in chloride and in sulphate solutions; the most important mass gain is recorded in sulphate solution.
- The mass variation of control mortar is comparable with that of PP fibres waste mortars, especially in chloride and in sulphate solutions, which confirmed the lower effect of this waste on the variation of mortar weight in these environments.
- Mortars immersed in sulphuric acid solution have showed a loss of their mass after an exposure of 180 days.
- PP fibres waste mortars immersed in different chemical solutions tested in this experimental work have showed a good flexural and compressive behaviour, compared to mortars without fibres.
- The used PP fibres waste has presented an excellent chemical behaviour and a high capacity to reduce the strength loss of mortars subjected to sulphate and acid attack.
- The X-ray diffraction confirmed the good behaviour of
mortar in sulphate and chloride solutions. It has showed that the degradation of mortar immersed in acid environment is caused by the formation of more gypsum product.

- No visual damage is observed on mortars exposed over one year to sodium chloride or magnesium sulphate solution with a concentration of 5%.
- Only mortars immersed during one year in 5% sulphuric acid solutions have showed a visual damage on their surface which is extended until approximately 300 μm depth.

The most important finding of this study is the success to fabricate a promotive and environmentally friendly composite (fibres mortar), based on available materials, with high mechanical performance and good chemical resistance. Another important finding is the participation to the protection of the environment and the human health by the recycling of some wastes. The use of waste provides many economical gains in construction projects. Technical gains are also provided by the reuse of certain waste, such the PP fibres waste obtained from domestic plastic sweeps’ fabrication as fibrous reinforcement for mortar and concrete. The results obtained from this investigation are very interesting from the point of view of durability.

References


De Schutter, G. (2012), Damage to Concrete Structures, CRC Press, Taylor & Francis Group, Boca Raton, USA.


of cement composition, sand type and exposure temperature”, 
https://doi.org/10.1016/j.conbuildmat.2012.06.048.
mechanical properties of fiber-reinforced concretes at low-
Stakne, K., Smole, M.S., Kleinschek, K.S., Jaroschuk, A. and
https://doi.org/10.1023/A:1023776030473.
Sun, Z. and Xu, Q. (2009), “Microscopic, physical and mechanical
https://doi.org/10.1016/j.msea.2009.07.056.
“Characteristics of concrete cracks and their influence on
https://doi.org/10.1016/j.conbuildmat.2016.01.002.
Wegjan, F.M. (2010), “Effect of seawater for mixing and curing on
Wittmann, F.H. and Zhao, T. (2012), “Knowledge of
microstructure of concrete for the design of durable reinforced
concrete structures”, Second International Conference on
Microstructural-related Durability of Cementitious Composites,
Amsterdam, Netherlands, April.
on the deterioration and approaches to enhance the durability of
cement in the marine environment”, Cement Concrete Compos.,
113, 103695.
magnesium sulfate concentration on the durability of
cement mortar with and without fly ash”, Compos. Part B: Eng,
Yin, S., Tuladhar, R., Shi, F., Combe, M., Collister, T. and
A review”, Constr. Build. Mater., 93, 180-188.
https://doi.org/10.1016/j.conbuildmat.2015.05.105.
the expansion of concrete under attack of sulfate and sulfate–
https://doi.org/10.1016/j.conbuildmat.2012.05.003.
durability of concrete composite containing fly ash and silica
“Understanding of the deterioration characteristic of concrete
exposed to external sulfate attack: Insight into mesoscopic pore

CC