INFLUENCE OF NATURAL POZZOLAN ON THE BEHAVIOR OF SELF-COMPACTING CONCRETE UNDER SULPHURIC AND HYDROCHLORIC ACID ATTACKS, COMPARATIVE STUDY

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الخلاصـه:

نظراً لأضراره الكبيرة على الهياكل الخراسانية في كل من المناطق الحضرية والصناعية يمثل هجوم العناصر الحمضية موضوعاً ذا أهمية متزايدة. إن نوعية المادة المعدنية المضافة من أهم العوامل التي تؤثر في مقاومة الخرسانة ذاتية الرص (SCC) في البيئة العدوانيه . ونادراً ما يُستخدم البوزولان ذو المصادر الطبيعية في الجزائر في (SCC) نظراً لعدم وجود دراسة مستفيضة عن خصائصها.

يتجه الهدف من هذه الدراسة إلى مقارنة سلوك (SCC) الذي يحتوي على البوزولان الطبيعي و (SCC) الذي يحتوي على الرماد المتطاير والحجر الجيري في وسط حمض الكبريتيك وحمض الهيدرو كلوريك. وقد تم - لهذا الغرض - إعداد اثنتي عشرة صيغة مختلفة تشمل ثلاثة فصول (30، 50 و 70 MPa) حيث تُغطس العينات بعد 28 يوما من المعالجة في محلول حمض الهيدرو كلوريك وحمض الكبريتيك لمدة 12 أسبوعا. وقد سجلنا - ضمن فترة الاختبار - التغيرات التي طرأت على الوزن وفقدان المقاومة لقوة الضغط. وقد استُخدم المجهر الكتروني الماسح (SEM)، وجهاز حيود الأشعة السينية (XRD) لفهم أفضل لآلية فقدان الوزن والمقاومة لكل نوع من أنواع الخرسانة المدروسة.

وتؤكد النتائج - بغض النظر عن خصائصها الاقتصادية - أن استخدام البوزولان الطبيعي الجزائري يساهم في تحسين مقاومة (SCC) ضد أخطار حامض الكبريتيك وحامض الهيدروكلوريك.

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ABSTRACT

Acidic attack is a topic of increasing significance, owing to the spread of damage to concrete structures in both urban and industrial areas. Mineral addition type is an important factor affecting performance of Self-Compacting Concrete (SCC) in an aggressive environment. Pozzolan from natural sources in Algeria is rarely used in SCC due to the absence of a thorough study of its properties. The goal of this study was to compare the hydrochloric and sulphuric acid behaviors of a SCC-containing Algerian natural pozzolan with SCC-containing fly ash and limestone filler additions. For this purpose, twelve formulations were prepared with three different strength classes (30, 50, and 70 MPa). After 28 days of curing, the samples were immersed in hydrochloric sulfuric acid solutions for a period of 12 weeks. The changes in mass loss and compressive strength loss for each acid solution within the test period were recorded. The Scanning Electron Microscope (SEM) and XRD analysis were used to better understand the mechanism of deterioration of each type of concrete. In spite of their economical properties, the results confirm that the use of Algerian natural pozzolan contributes to the improvement of resistance of SCC under sulphuric and hydrochloric acid attacks.

Key words: self-compacting concrete, natural pozzolan, mass loss, compressive strength loss, sulphuric acid, hydrochloric acid

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1. INTRODUCTION

Self-Compacting Concrete (SCC) describes a concrete with the capacity to compact itself only by means of its own weight without the requirement of vibration. It fills all recesses, reinforcement spaces, and voids, even in highly-reinforced concrete members and flows free of segregation nearly to balance level. Flowing in the formwork, SCC is able to deaerate almost completely.

SCC may contribute to a significant improvement in the quality of concrete structures and open up new fields in the application of concrete [1].

Due to its specific properties, the study of SCC represents an area of research that has strong potential development [2]. However, in spite of the great interest of researchers in this new material, SCC has not yet gained universal acceptance as a construction material and its application is still limited. Because of its higher binder and chemical admixture content, SCC is usually associated with a 20–50% higher material cost compared to that of ordinary concrete with comparable compressive strength [4]. Martin [4] found that depending on the fly ash content of SCC, its material cost is 10–17% higher than that of ordinary concrete.

The development of an economical SCC with interesting properties in the fresh and hardened state will certainly help and encourage the use of this material in the construction industry [5].

The use of natural and economical materials seems to be one of the possible solutions for the future. In this paper, the natural pozzolan of west Algeria is considered.

The objectives of this work are the valorization of the locally natural pozzolan in addition to an economical SCC, and the evaluation of its influence on the resistance of SCC in acid mediums.

The choice of this parameter of durability is justified by its importance in the evaluation of the service life of concrete structures [6].

Concrete structure, in general, is regularly subjected to aggressive environmental conditions. Due to its interactions with external influences, the mechanical and physical properties of concrete may be threatened and lost [7].

Sulphuric acid (H_2SO_4) and hydrochloric acid (HCl) are classified as the most aggressive natural threats [8]. Their spectrum media is wide. Usually they originate from industrial processes, but they can even be due to urban activity. Significant quantities of acids are present in sewage systems [9]. The acidic attack is affected by the processes of decomposition and leaching of the constituents of cement paste [8]. The very important volume of the addition in the paste of the SCC can be influenced positively or negatively by their resistances to acid aggressions [9,10]. In this present study, we evaluate the effect of natural pozzolan, limestone filler, and fly ash on the behavior of the SCC against sulphuric and hydrochloric acid attacks.

2. MATERIALS

2.1. Cement

Ordinary Portland cement (ASTM type I) is used. The chemical analysis of this cement and the mineralogical composition of clinker are presented in Table 1.

2.2. Aggregate

The sand used in this study is siliceous round of 4 mm maximum aggregate size, with a specific gravity and water absorption of 2.6 and 1.2%, respectively. Crushed siliceous limestone coarse aggregates with a nominal size of 12.5 mm, specific gravity of 2.66, and water absorption of 0.5% were used for the concrete samples.

2.3. Addition

Three types of additions are used in this study:

- A natural pozzolan from a volcanic deposit in the western region of Algeria.

-A limestone filler characterized by its great fineness.

- A silico-allumineuse fly ash class F.

The particle size distributions of the mineral additions are shown in Figure 1.

Table 1 describes the various physical and chemical characteristics of each addition.

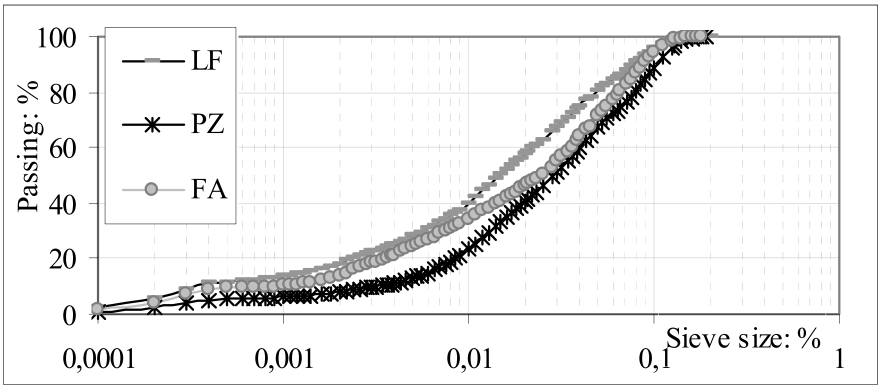

Figure 1. Particle size distributions of the filler additions

Table 1. Chemical Composition and Physical Properties of Cement and Additions

Composition %	Cement	Limestone filler	Fly ash	Natural Pozzolan
SiO_2	21	0.4	49.6	45.67
$CaCO_3$	-	98.5	-	-
CaO	68	-	3.00	8.98
Al_2O_3	5.81	-	23.8	15.1
Fe_2O_3	3.26	-	17	10.14
MgO	1.2	-	1.3	3.45
SO_3	2.51	0.074	0.1	0.19
Specific gravity	3.13	2.70	2.2	2.61
fineness Blaine (m^2/kg)	380	406	384	365.6
Activity factor i_{28}	-	0.75	0.79	0.81

2.4. Superplasticizer

In this study, we used acrylique copolymère superplasticizer. Its density was 1.06 and its chloride ion content was below 0.1%, characterized by its great rheology duration and its resistance to segregation. It enabled us to realize the recommended tests in the fresh state without using a retarded setting.

3. EXPERIMENTAL PROGRAM

3.1. Mixtures

The formulation of the SCC is determined by using the “BétonLab.Pro” software [11]. We took into account the various relative data of the used materials. Twelve formulations were prepared, thus covering three various strength classes (30, 50, and more than 70 MPa) and four types of concretes (Ordinary Vibrated Concrete (OVC), SCC with limestone filler addition (SCC LF), SCC with natural pozzolan (SCC PZ), and SCC with fly ash (SCC FA)) .

We chose a fixed content of the binder (cement + mineral additions) equal to 520 kg/m^3. In each strength class, the concretes are formulated starting from the same components, with the same granular skeleton and constant Water/Binder (W/B) ratio. The comparison is then carried out with the same mechanical strength.

The quality of fresh concrete was checked according to the tests recommended by the AFGC [12]. Superplasticizer was used to obtain a slump flow as close as possible to 66±1cm for all the SCC and a slump of 6 cm for the OVC.

Table 2 shows the mixture proportions of the concretes developed in this study

Table 2. Mixture Proportions of the Concretes Investigated

Concrete \ Composition (kg/m^3)	C	LF	PZ	FA	S (0/4)	G4/6	G6/12	w (l)	S-P (l)	W/B
OVC 70	450	-	-	-	747	154	900	176	2.9	0.39
SCC70 LF	450	70	-	-	840	171	687	183	9.7	0.4
SCC70 PZ	450	-	70	-	832	170	681	188	10.5	0.4
SCC70 FA	450	-	-	70	826	168	676	188	11.50	0.4
OVC50	350	-	-	-	777	159	936	182	0.9	0.52
SCC50 LF	350	170		-	814	166	666	198	6.7	0.52
SCC50 PZ	350	-	170	-	790	161	647	214	6.0	0.52
SCC50 FA	350	-	-	170	775	158	634	214	4.0	0,52
OVC30	260	-	-	-	830	171	1000	183	0.9	0.7
SCC30 LF	260	260	-	-	807	164	661	199	2.9	0.7
SCC30 PZ	260	-	260	-	782	159	640	215	3.7	0.7
SCC30 FA	260	-	-	260	758	155	620	215	3.8	0.7

3.2. Physico-Chemical Testing of Concretes

After 28 days of curing at 20°C and 95% relative humidity, the initial mass of the cubic specimens of 10x10x10 cm^3 was determined before immerging in the acid solutions.

Sulphuric and hydrochloric acid solutions with initial concentrations of 5% by volume were prepared in acid-resistant tanks. Replicates of specimens from each mixture were kept continuously immersed in the various acid solutions for 12 weeks as recommended by ASTM C 267 [13]. During the test period, the cubic specimens were removed weekly from solutions, rinsed with tap water, without brushing, and left to dry for 30 min before weighing and visual inspection. The solution was renewed with each new weighing to maintain constant concentration [14]. Cumulative mass change (MCt) for each specimen was determined as follows:

$$MCt, \% = [(M_0-M_i)/M_0]*100 \quad (1)$$

where M_0 is the initial mass before exposure to acid solutions (kg) and Mi (i=1,2,3,…,12) is the mass after i weeks of exposure to acid solution (kg).

After 12 weeks [13], the specimens were capped and tested for residual compressive strength based on the original cross-sectional area. The percentage of strength change was calculated in the same way as the mass change equation. After compression testing, scanning electron microscopy (SEM) and XRD analysis were conducted on selected surface fractures to investigate the damage mechanisms.

4. RESULTS AND DISCUSSION

4.1. Concrete Properties in Fresh and Hardened State

The first results as given in Table 3 concern the fresh concrete properties. Generally, the SCC mixtures present good workability (slump-flow equal to 66±1cm, L-box results ranged between 0.80 and 0.89, and flow time values between 5.8 and 7 s). The fresh SCC properties complied with these AFGC recommendations [12].

Compressive strength results shown in Table 3 correspond to the average values obtained on at least three cylindrical samples (Ø 11× 22 cm). The results show very similar values for each strength class, which confirms the relative data with the equivalent binder.

Table 3. Concrete Properties in Fresh and Hardened States

Properties	OVC 70	SCC 70 LF	SCC70 PZ	SCC70 FA	OVC50	SCC50 LF	SCC 50 PZ	SCC50 FA	OVC30	SCC30 LF	SCC30 PZ	SCC30 FA
Slump (cm)	6	-	-	-	6.4	-	-	-	6	-	-	-
Slump flow (cm)	-	67	67	66	-	65	67	65	-	66	67	65
L-box H_2/H_1	-	0.86	0.85	0.83	-	0.80	0.85	0.83	-	0.86	0.89	0.82
V-Funnel (s)	-	5.8	6	7	-	6	6.1	6.2	-	5.8	6.4	6.8
Air content (%)	3.5	2.5	2.2	2	3.9	2.8	3	3.2	4.4	2.9	2.3	2.2
Rc 28 jours (MPa)	69.9	71.8	71.2	70.8	48.7	51.1	50.9	50.3	29.5	32.5	31.1	30.9

4.2. The H_2SO_4 Attack

The results of mass loss for specimens exposed to 5% sulphuric acid solutions are shown in Figures 2, 3, and 4.

From these results, we observe the continuous loss of the mass for all the studied concretes (from the first week until the twelfth). After one week, the results show the same tendency of deterioration, particularly of the 70 and 50 MPa classes (the difference is <1%). After 12 weeks, the difference became remarkable.

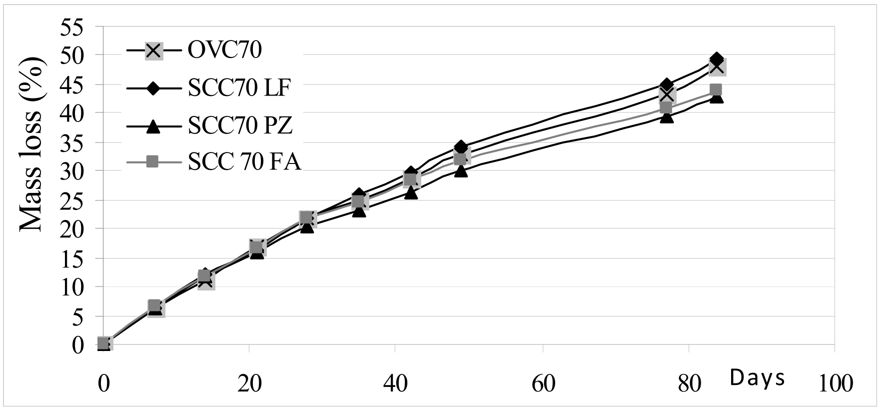

Figure 2. Mass loss of concrete specimens due to immersion in 5% H_2SO_4 solution (70 MPa strength class)

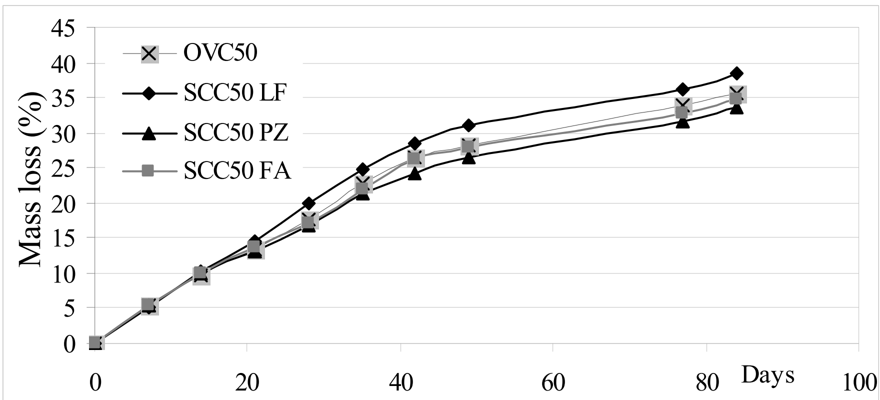

Figure 3. Mass loss of concrete specimens due to immersion in 5% H_2SO_4 solution (50 MPa strength class)

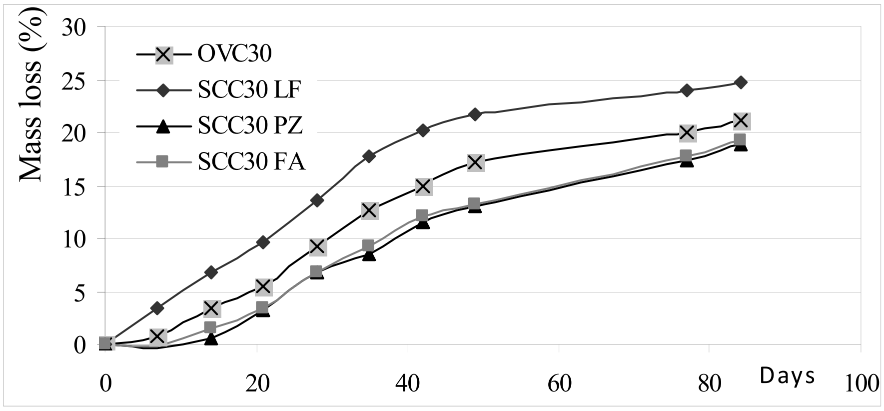

Figure 4. Mass loss of concrete specimens due to immersion in 5% H_2SO_4 solution (30 MPa strength class)

The SCC mixtures with limestone filler addition present the greatest mass loss in comparison with the other concretes (49% for SCC70 LF, 39% for SCC50 LF, and 25% for SCC30 LF).

The SCC mixtures with natural pozzolan addition are more resistant to sulphuric acid attack, with a mass loss equal to 43% for SCC70 PZ, 34% for SCC50 PZ, and 18% for SCC30 PZ. The difference between the results of SCC mixtures with natural pozzolan and SCC with fly ash addition is very small (<3%).

The comparison between the result of SCC and OVC is related to the nature of the addition. The SCC prepared with natural pozzolan addition and fly ash addition, are always more resistant to sulphuric acid than the OVC. With limestone filler, the SCC is still, in general, less resistant.

Moreover the higher strength concretes (70 MPa strength class) show a higher weight loss, particularly at higher exposure times. This was probably due to the fact that the sulphuric acid attack as it is known is primarily related to the actual cement contents in these concretes. Obviously, the lower strength concretes show better resistance against this acid attack. Dinakar *et al.* [10] studied the effect of the incorporation of a high percentage of fly ash on the durability of SCC in acid medias. They linked the process of deterioration to the low amount of reactive compounds like $Ca(OH)_2$ at lower levels of cement content. Similar conclusions were reported earlier by Fattuhi and Hughes [15,10]. They subjected cement paste and concrete to 2% H_2SO_4 attack in a continuously flowing hydraulic channel for 50 days and assessed the performance using the weight loss with time. They concluded that the H_2SO_4 attack decreased with the decrease in cement content.

A visual inspection of specimens as shown in Figure 5 reflected the deterioration of the samples, particularly for the concretes with high cement content (450 kg/m^3). These concretes kept, more or less, their cubic forms, but their dimensions decreased considerably. For example, the SCC70 LF lost 2 to 3cm on all sides.

Figure 5. Deterioration of specimens after 12 weeks of immersion in 5% H_2SO_4 solution

According to these results, we can note that all mixtures containing limestone filler had a higher neutralization capacity (acid consumption) due to the presence of calcium carbonate, but with the high fineness (406 m^2/kg) of the limestone filler used in the present study, the kinetics of the reaction with sulphuric acid also accelerate the deterioration of SCC. Similarly, Bassuoni *et al.* [9] reported an increase in the mass loss of the SCC made with finely ground limestone filler immersed in a sulphuric acid solution. They also found that, depending on the nature of limestone fillers and the concentration of acid solutions, increasing the percentage of limestone fillers increases the mass loss up to 60%.

For the SCC mixtures with pozzolanic addition, the pozzolanic reaction increased the formation of C–S–H. The last one is characterized by its progressive decalcification to form other hydrates such as ettringite and monosulfoaluminate, while the portlandite passes totally in solution. Torri and Kawamura [16] also reported similar observations. They examined the effect of 2% H_2SO_4 solution on fly ash and silica fume mortars. Fly ash mortars with 70% replacement were studied for 3 years. They observed that the effect of H_2SO_4 attack decreases with increasing fly ash replacement.

4.3. The HCl Attack

Figures 6, 7, and 8 show the results of mass change for specimens exposed to 5% hydrochloric acid solutions.

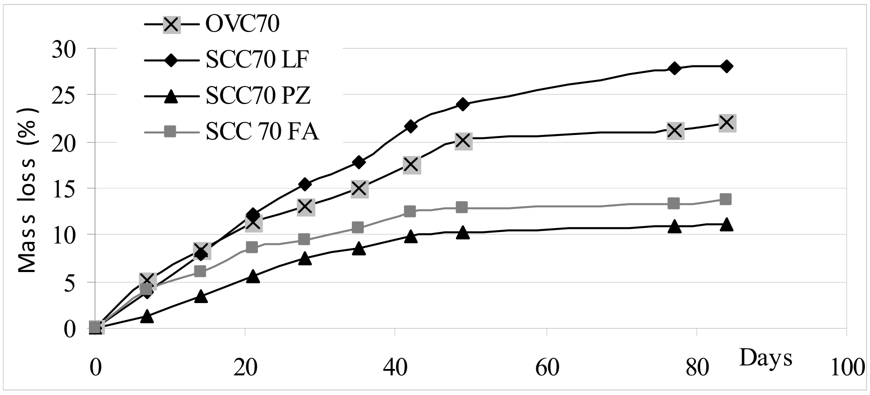

Figure 6. Mass loss of concrete specimens due to immersion in 5% HCl solution (70 MPa strength class)

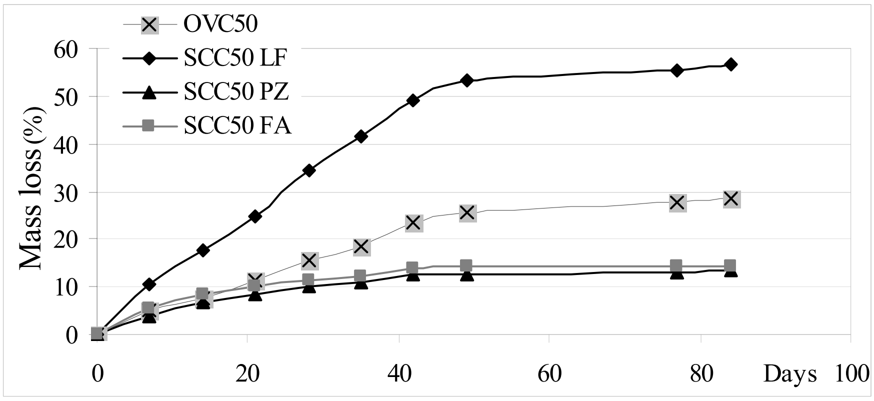

Figure 7. Mass loss of concrete specimens due to immersion in 5% HCl solution (50 MPa strength class)

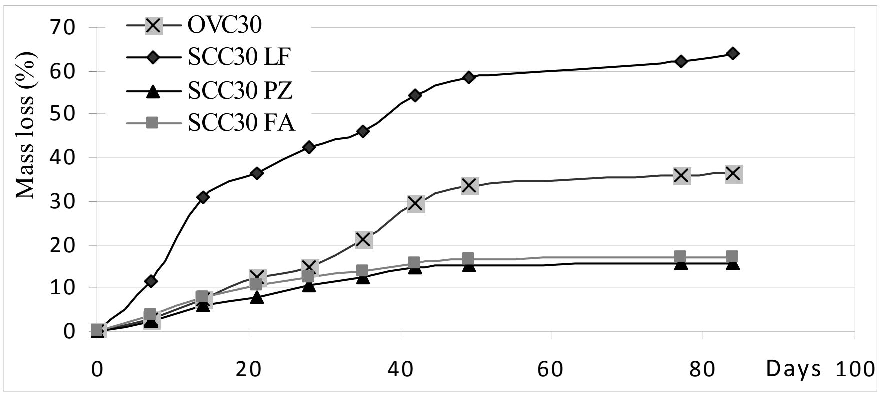

Figure 8. Mass loss of concrete specimens due to immersion in 5% HCl solution (30 MPa strength class)

When comparing the concrete at the same strength class, we notice that the SCC mixtures with limestone filler addition present the largest mass loss. The SCC mixtures with natural pozzolan addition present the lowest mass loss. The incorporation of natural pozzolan in the paste of SCC reduces the capillary pores by a new C-S-H gel formed from the pozzolanic activity and portlandite hydrate resulting from the hydration of cement. The formation of calcium chlorides, salts very soluble in water, due to the reaction of hydrochloric acid with cement, is more important in the presence of limestone fillers. The presence of a high content of calcium carbonate ($CaCO_3$) increases the capacity of limestone fillers to consume more hydrochloric acid. The very high fineness of the limestone fillers also increases the aggressiveness of the acid to leach the paste quickly, particularly on the exposed specimen's surface.

Visually we clearly see the degraded state of the different samples after 12 weeks of immersion in 5% HCl solutions (Figure 9). The SCC30 LF and the SCC50 LF have totally lost their cubic form (loss of more than half of the sample). The OVC30 and OVC50 also have remarkably lost their forms, while the other concretes have undergone a small change. These results are in good agreement with other studies in the literature showing the beneficial effect of silica fume and fly ash addition on the resistance of cement paste to leaching [17,18].

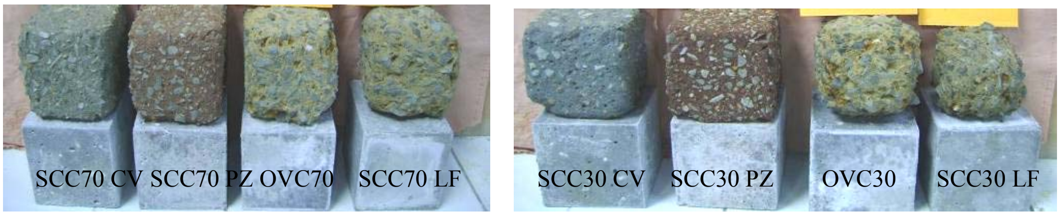

Figure 9. Deterioration of specimens after 12 weeks of immersion in 5% HCl solution

4.4. Comparison Between the Kinetics of Sulphuric and Hydrochloric Acid Attacks

By comparing the aggressiveness of sulphuric acid medium and hydrochloric acid medium, we conclude that, for the same concentration, the extent of deterioration is different. For the SCC mixtures with limestone filler addition, the loss of weight is lower in the sulphuric acid solutions. The other concretes present more mass loss in the sulphuric acid than in the hydrochloric acid solutions. For the same type of concrete, degradation mechanisms due to H_2SO_4 attack and HCl attack are different. Sulphuric acid leached the layers of the paste in exposed surface, while hydrochloric acid penetrates inwards the concrete by the interval of porosity.

4.5. Scanning Electron Microscope Observations (SEM)

Figure 10 shows SEM photographs after 12 weeks of immersion in 5% sulphuric acid solution.

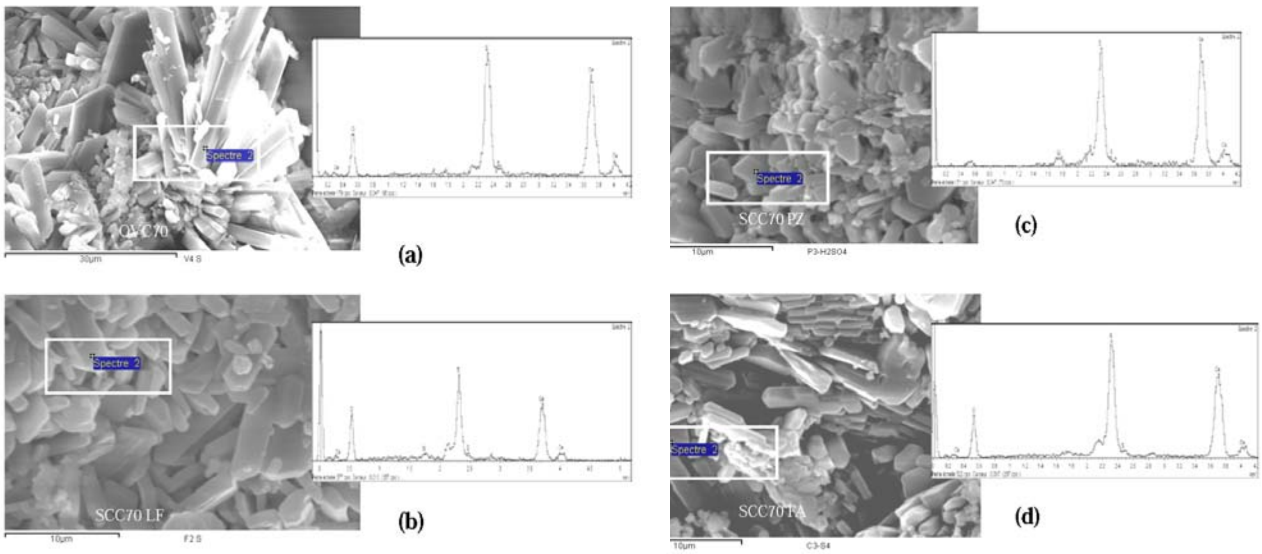

Figure 10. SEM and EDAX data for concretes specimens after 12 weeks of exposure to sulphuric acid: (a) OVC70, (b) SCC70 LF, (c) SCC70 PZ and (d) SCC70 FA

Micrographs of concrete specimens exposed to sulphuric acid for 12 weeks indicate that the surface of concrete underwent significant deterioration since it is highly porous and rich in weak crystalline structures (calcium sulphate)

In comparison, the different photographs show a strong densely fibrous structure for the SSC30 LF (Figure 10 (b)) and OVC30 (Figure 10 (a)), which confirms the strong leaching of the cement paste on the surface of this concrete. The EDAX data clearly show the dominant elements of calcium and sulphur and, hence, the white products are gypsum. The abundance of gypsum crystals on the surface facilitates their dissolution in water, which explains the mechanism of deterioration of the concrete after immersion in sulphuric acid solutions. This conclusion is in agreement with observations of Yamanaka *et al.* [19] and Bassuoni *et al.* [9]. These authors noticed that there was a successive decomposition of concrete specimens starting from the exposed surface and moving inwards. Bassuoni *et al.* [9] found that about 10 mm from the surface of concrete exposed to sulfuric attack, the micrographs of (SEM) indicate a densely fibrous C-S-H structure, which explains why the concrete core was still sound.

4.6. X-ray Diffraction Analysis (XRD)

Figure 11 presents the x-ray diffraction analysis for each type of concrete studied in this paper.

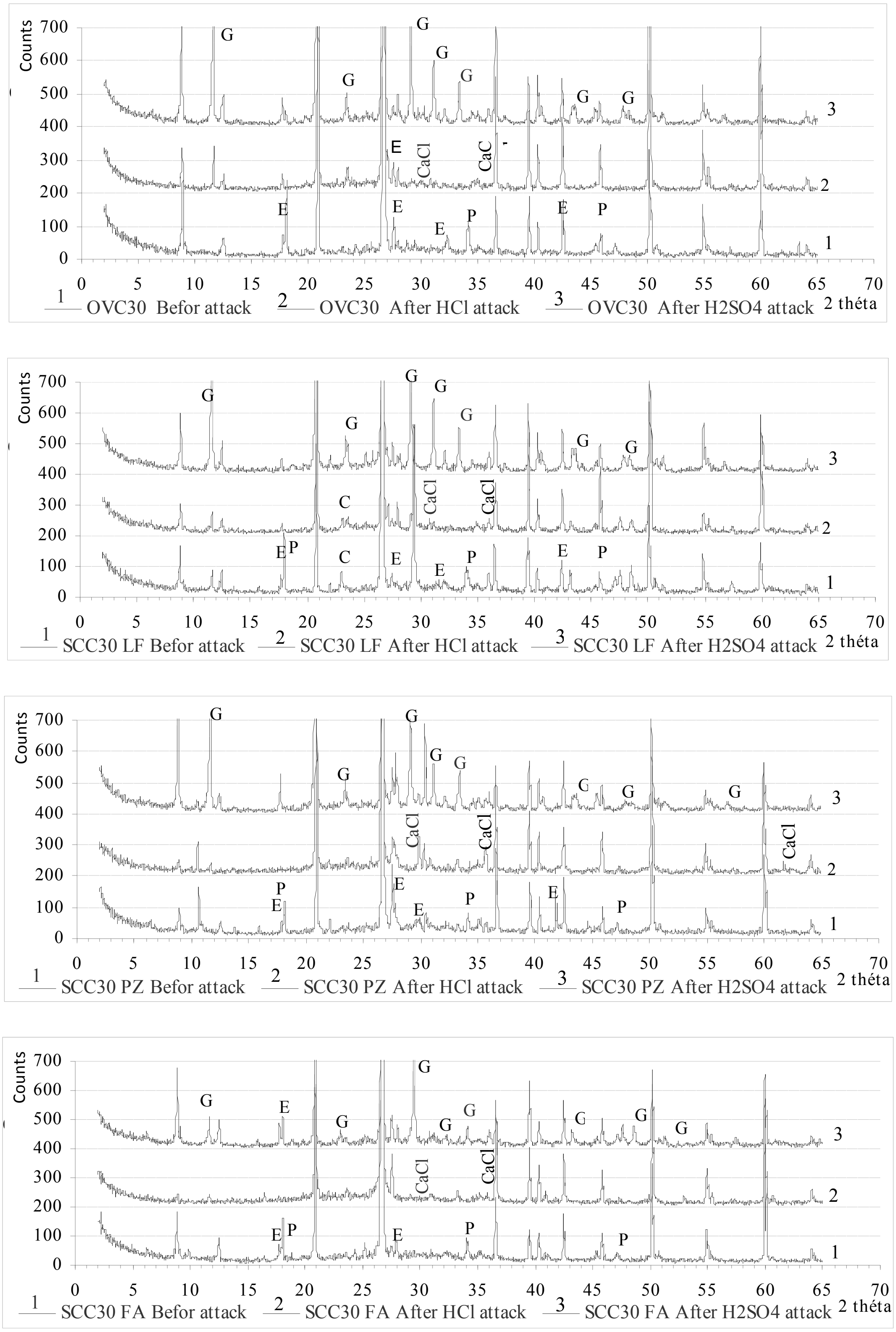

Figure 11. The X-ray diffraction spectra before attack, after HCl and after H_2SO_4 attack G: Gypse, P: Portlandite, E: Etringite

The superposition of the 3 various spectra (initial state, after HCl attack, and after H_2SO_4 attack) confirms the appearance of a great quantity of gypsum (G) on the specimens which were exposed to the sulphuric acid, and a trace of calcium chloride (CaCl) for the specimens which were exposed to hydrochloric acid.

The low quantity of calcium chloride is due to its high solubility in water. The washing of each specimen after the period of immersion makes its surface almost empty of this salt.

4.7. The Relation Between the Compressive Strength Loss and the Mass Loss

The compressive strength of SCC30 LF, SCC50 LF, and OVC30 were not measured, due to the severe deterioration of samples after 12 weeks of HCl attack.

Figures 12 and 13 illustrate the relationship between compressive strength loss and mass loss as a percentage of their initial values before exposure.

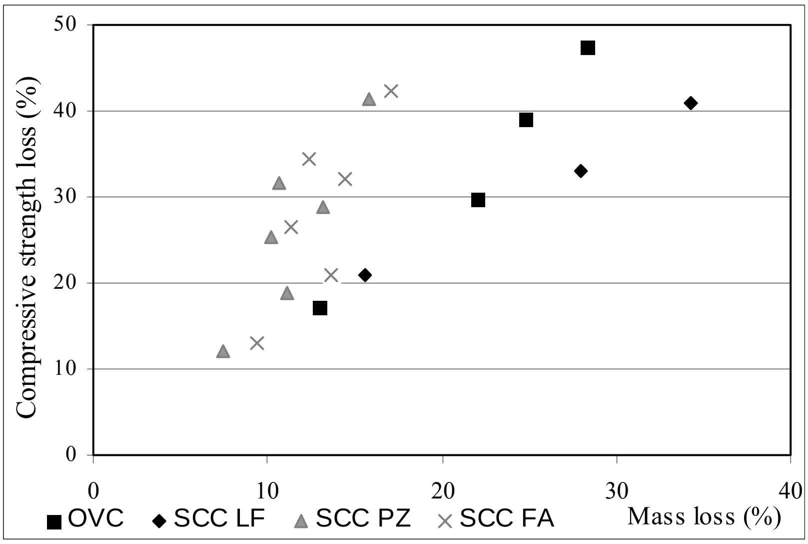

Figure 12. Compressive strength loss versus mass loss after 12 weeks of exposure to HCl solution

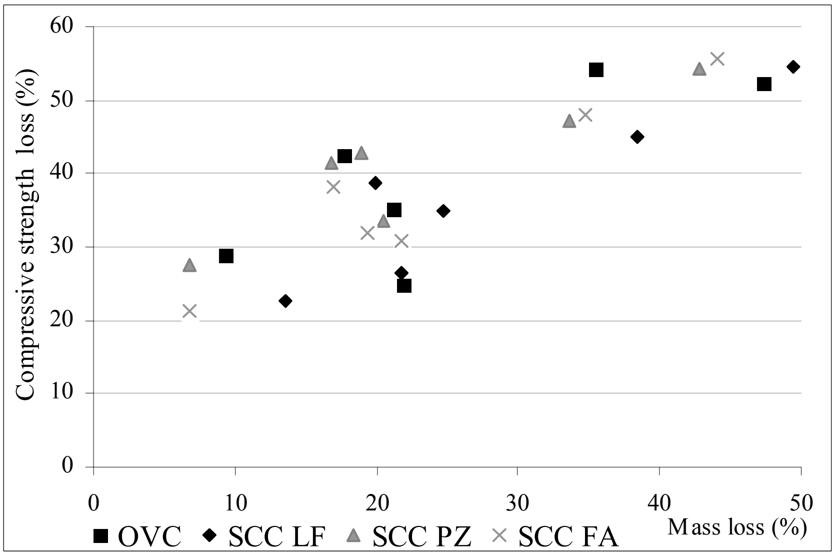

Figure 13. Compressive strength loss versus mass loss after 12 weeks of exposure to H_2SO_4 solution

The results reveal that there is not a direct relationship between the compressive strength loss and the mass loss for the concrete specimens immersed in HCl solutions (Figure 12). In the same way, for the results of the sulphuric acid, represented in Figure 13, the external state of the degradation of the samples after exposure to the sulphuric acid attack is not a good indicator of their compressive strength; in fact, there is a divergence between the loss of mass and the loss of the compressive strength. The observed trend of the compressive strength change after 12 weeks of exposure to HCl and H_2SO_4 solutions is affected by multiple factors. After exposure to acid solutions, concrete specimens had a softened surface zone underlain by a sound part, which represents the bulk cross-section of specimens. The observed compressive strength reduction after exposure depends on the ratio of deteriorated-to-sound cross-section [20]. The existence of a deteriorated (protective) zone, especially in specimens immersed in HCl solution (≥10mm), can decrease mass loss of specimens due to limiting acidic diffusion into the cementitious matrix. Conversely, the occurrence of this zone can increase strength loss results since it has weak mechanical properties, as shown by different studies in the literature [21,22]. Moreover, after exposure to sulphuric acid solutions, the change in aspect ratio and variability of geometry (diameter) across the height of specimens may involve load eccentricity and non-uniform stress distribution during compression testing, which had an effect on the reliability of the determined crushing load.

Previous studies (Chang *et al.* [23] and De Belie *et al.* [24]) have even found that there is an increase in compressive strength in some samples of the SCC after their exposures to the attack sulphuric acid, although there is a significant loss of weight.

5. CONCLUSION

Based on the experimental results reported in this paper, the following conclusions can be made:

- The SCC with natural pozzolan addition has a better behavior against the aggressions of sulphuric acid and hydrochloric acid mediums in comparison with the SCC containing limestone filler, and quite similar behavior in comparison with the SCC- containing fly ash.

- The change in compressive strength was not a proper indicator of the surface deterioration of concrete exposed to sulphuric and hydrochloric acids environments. In spite of the great compressive strength, the increase of cement content increases the risk of the sulphuric acid attacks.

- For the same strength classes, the comparison between the SCC and OVC is related to the nature of addition. The SCC with natural pozzolan or fly ash addition has a better resistance in the acid mediums than the OVC. With a limestone filler addition, the SCC has a lower resistance than the OVC.

Finally, the incorporation of the Algerian natural pozzolan as addition in the SCC is very beneficial to the resistance of these concretes in sulphuric and hydrochloric acid mediums. However, the use of limestone filler in the hydrochloric acid environment seems to be not recommended.

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