Journal of Civil Engineering and Architecture 17 (2023) 343-357
doi: 10.17265/1934-7359/2023.07.004

Microstructure and Permeability of Concrete with Glass Powder Addition Preserved in the Sulphatic Environment

Rabah Chaïd
Materials and Environment Research Unit, University of Boumerdes, Algeria

Abstract: Most mechanical properties and durability of cementitious materials are related to the performance of the hydrated cement that coats the granular skeleton. In this paper, concrete is formulated by substituting 30% of cement by finely ground glass powder. The experimental study consists of investigating the effect of conservation in tap water or sulfate water on the performance of concrete. The mechanisms of concrete damage have been related to the development of the microstructure of the material. The degradations were observed using scanning electron microscope (SEM) and quantified by x-ray diffraction (XRD). In addition, chloride ions permeability and gas permeability tests were performed. The results have shown that glass powder present a pozzolanic activity and hence it affects favorably the microstructure of the paste which becomes denser and less permeable.

Key words: Concrete, glass, permeability, microstructure, sulphate.

1. Introduction

Different mineral additions are currently used in concrete. They are used as addition or substitution to cement. The use of these supplementary cementitious materials provides to concrete a denser matrix that will be more resistant to aggressive environments such as sulphates, chlorides and other aggressive agents. The fineness of mineral additions, their chemical composition, their structure (glassy or crystalline), their pozzolanic activity and their solubility in alkaline medium govern their cementing properties in concrete. Their content in amorphous phase is a key factor in the reactivity of the mineral additions [1].

Glass is obtained primarily from the fusion of silica sand (> 99% silica), and hence could be a pozzolanic material. As the use of glass increases steadily, glass waste increases accordingly. There are many alternatives for glass reuse. According to some studies [2], most methods of reuse require preliminary preparation such as milling and hence could not be well developed and are unable to absorb the large quantities of waste glass. An alternative sustainable solution could be the reuse of waste glass in concrete. Indeed, for economic and environmental reasons, the use of recycled glass in cement and concrete has attracted the interest of many municipalities which encouraged further studies [3].

The decrease in porosity through the reduction of the W/C ratio by adding super plasticizer that deflocculates cement grain and ensures sufficient workability, as well as the enlargement of their grading with the addition of ultrafine elements give a denser concrete and more resistant to chemically aggressive environments [4].

This paper reports on a part of an ongoing research project on the reuse of mineral waste materials as an addition to cement for the development of a sustainable concrete. This paper discusses mainly the results of an experimental study on the effect of finely crushed glass on the microstructure of concrete cured in three different environments: tap water and sulfate water. The evolution of the crystalline phases is analyzed by

Corresponding author: Rabah Chaïd, research fields: concrete.

X-ray diffraction and Electron Microscope (SEM). The microstructural study concerns both the surface layer and the internal structure of the specimens. In addition, the paper reports the results of chloride ion and oxygen permeability tests.

2. Matérials Used

2.1 Cement

The cement used was a local Portland cement type CEM II/B 42.5 with 18% limestone addition. The chemical and mineralogical compositions of the cement are summarized in table 1. Table 2 gives the mechanical characteristics of the cement.

2.2 Fine Aggregates

Modified sand (a mixture of 36.56% siliceous fine sand and 62.55 % coarse calcareous sands) was used. Table 3 summarizes the physico-mechanical characteristics of the two sands. It should be noted that sand with a high fineness modulus is preferred for the manufacture of high performance concrete (HPC) to reduce the amount of mixing water [5]. In this study, the fineness modulus of the used sand was Mf = 2.8.

2.3 Coarse aggregates

The coarse aggregates used are of calcareous origin and their physical and mechanical properties are presented in table 4. The maximum aggregates size adopted according to previous studies as 13 and 14 mm [6] and the ratio gravel/sand 1.5 to 2 [7]. The grading curves are given in Fig. 1.

2.4 Superplasticiser

The used superplasticiser was a modified polycarboxylate type MEDAFLOW 40HR. It is a chloride free light green liquid that can maintain workability for up to 1h30 min.

2.5 Glass Powder

The glass powder is obtained through grinding waste glass. The fineness of the glass was assured by passing it through an 80 µm sieve. The physical and chemical characteristics of the powder are given in tables 5 and 6.

The grain size of the glass powder was determined using laser granulometry (CILAS 1 180 type) and is given in figure 2. It could be seen that the grading is

Table 1 Chemical and mineral composition of the cement used.

Elements	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	IR	Free CaO	LOI
%	60.78	18.13	4.42	3.03	1.84	2.34	0.64	0.13	0.20	1.00	8.36
Minerals	C_3S		C_2S		C_3A		C_4AF				
%	57		19		05		13				

Table 2 Physical and mechanical characteristics of the cement used.

Physical characteirstics	Intial setting time	2h30
	Final setting time	4h00
	Density	3.1 g/cm^3
	Fineness (SSB)	3555 cm^2/g
	Heat of hydration	457 (j/g)
Mechanical characteristics	Age (days)	Compressive strength (MPa)
	7	36.78
	14	41.07
	28	45.07

Table 3 Properties of the used sands.

Type	Ma (kg/l)	Ms (g/cm^3)	Sand equivalent ESP (%)	Fineness modulus Mf
Boussada Sand	1.48	2.66	68	0.96
Baghlia Sand	1.53	2.73	73.2	3.86

Table 4 Coarse aggregates characteristics.

Types	Ma (kg/l)	Ms (g/cm^3)	Los Angeles
Gravel 3/8	1.36	2.63	26.70
Gravel 8/15	1.45	2.63	25.5

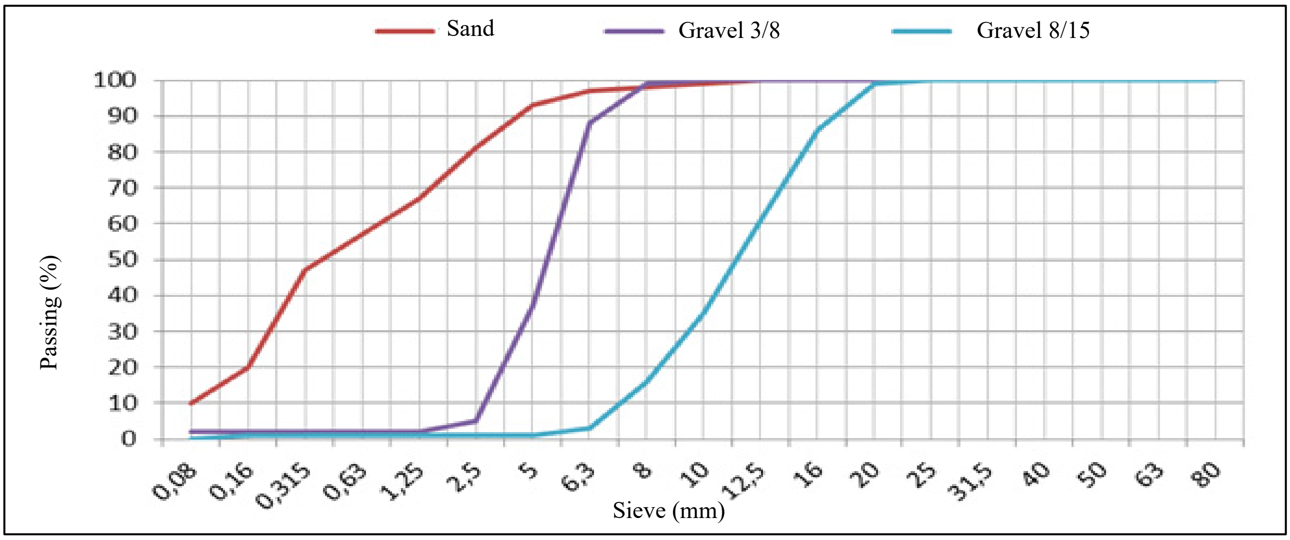

Fig. 1 Grading curves of the aggregates used.

Tablea 5 Chemical composition of the glass powder

Elements	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	TiO_2	PAF
%	72.8	0.98	0.55	9.7	1.76	0.25	0.43	12.8	0.01	0.04	0.79

Table 6 Physical characteristics of the glass powder.

Density (g/cm^3)	2.5
Specific surface (cm^2/g)	8000
Color	white grey
Pozzolanic activity	28.5 g [$Ca(OH)_2$/g (PV)]

very fine with more than 50% passing 8 µm and approximately 90% of the particles are less than 25 µm in diameter. Based on the coefficient of uniformity (Cu), which it could be concluded that the crushed waste glass is characterized by a very tight grading.

The absence of picks in the diffraction diagram of the glass powder (Fig. 3) confirms its amorphous state and hence its pozzolanic activity (Fig. 4).

3. Experimental Procedure

3.1 Concrete Composition

The concrete mixes with and without glass powder used in the experimental program are reported in table 6. It is worth noting that the percentage of addition of powdered glass (PG) and superplasticizer were adopted from earlier studies and were respectively 30% and 2% of the weight of cement.

The following notation was adopted:

- For concrete: BR for reference concrete without addition, BPV concrete with glass powder
- For paste: PR: Reference paste (without additions); PPV: paste with glass powder
- For curing environments: W: Tap water and SFW: sulfate water.

3.2 XRD and SEM Analysis

Concrete specimens are kept in a curing room (20 ° C, 95% RH) for 24 h. They are then cured either in tap water, water with 5% of gypsum or seawater for 7 days, 28 days or three months. The evolution of the different crystalline phases present in the concrete or newly formed is analyzed by x-ray diffraction using a diffractometer type Philips PW3710, with a copper anticathode and a Nickel filter (Cu Kα, λ = 0.154nm). The measurement is done by the step-to-step method (step of 0.02 ° 2θ).

In addition to XRD analysis, SEM analysis were also performed using an electron microscope type (Jeol conventional JSM 6400) used for the observation of dry samples that are treated superficially by palladium gold and analyzed by an Oxford Link Isis energy dispersive spectrometer.

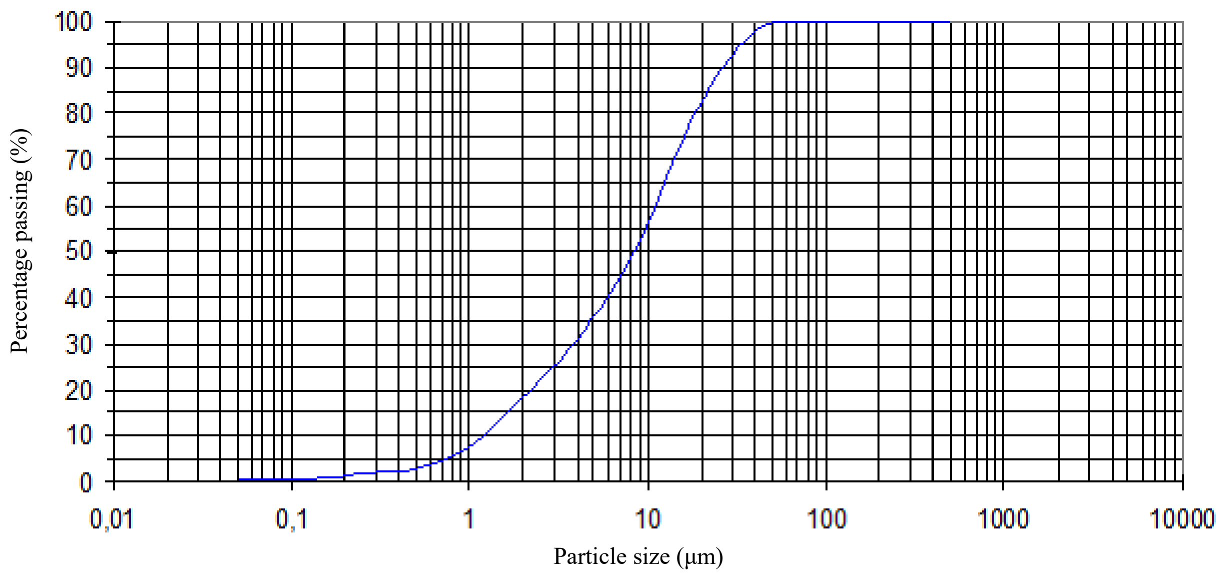

Fig. 2 Grading curve of the glass powder.

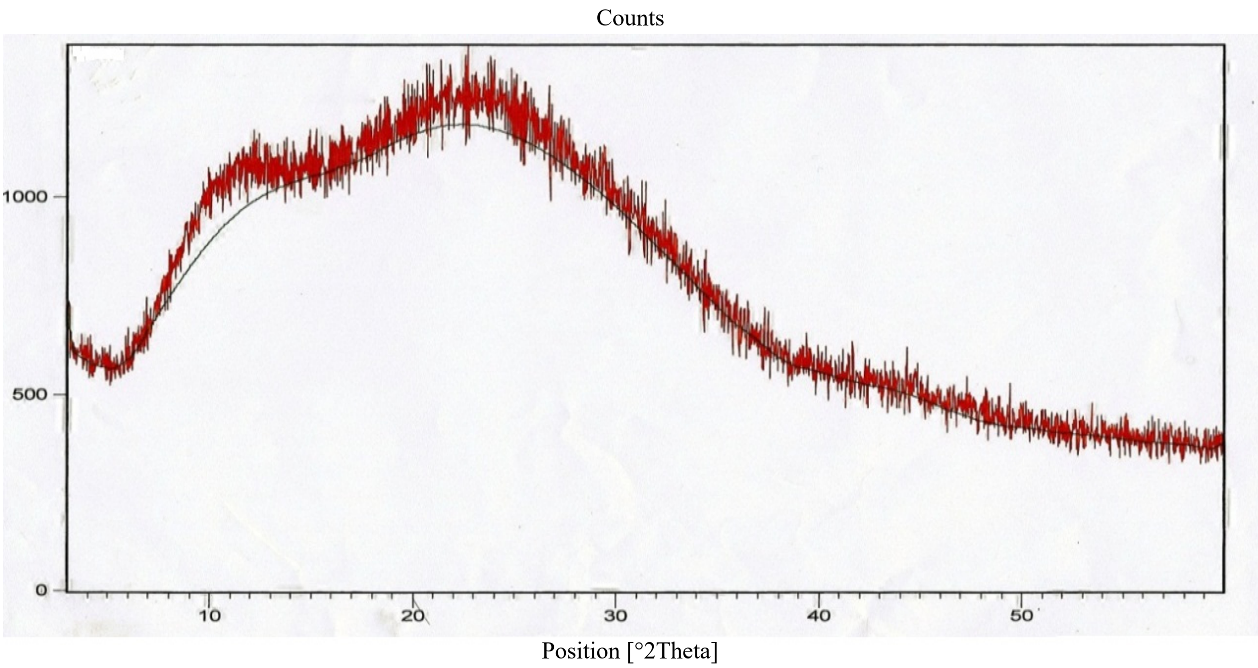

Fig. 3 X-ray diffraction of the glass powder.

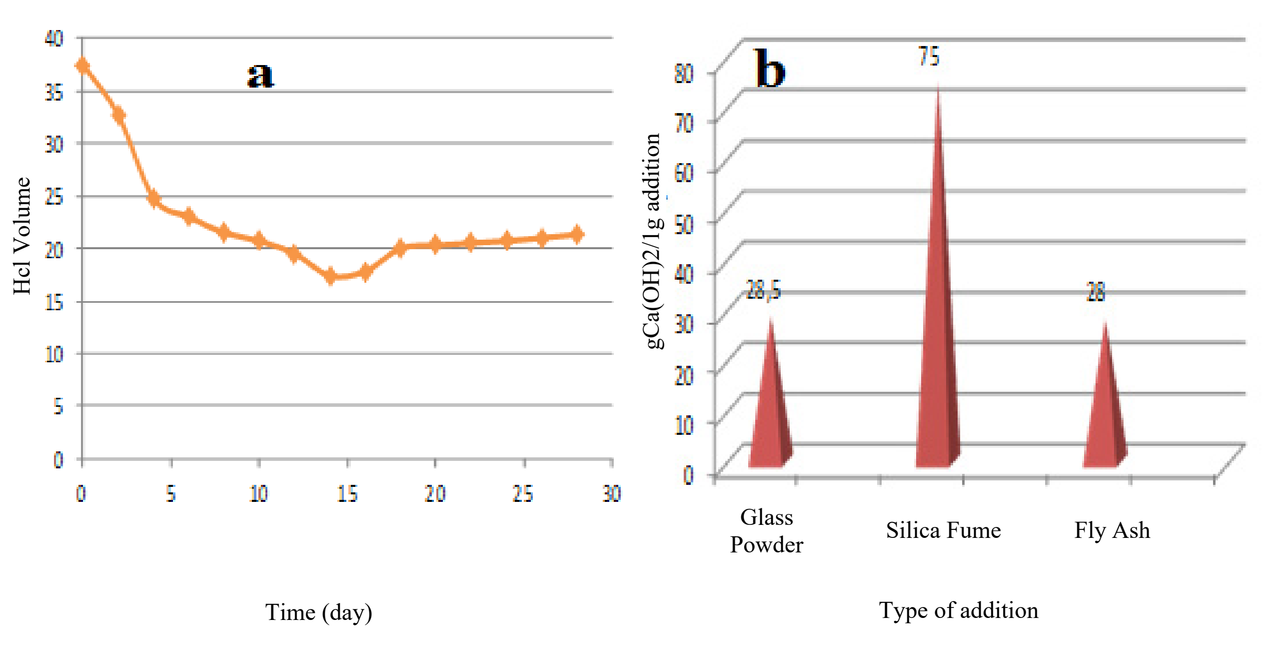

Fig. 4 a) Volume of consumed HCl; b) Pozzolanic activity of the glass powder.

Table 6 Concrete mixes.

Constituents	Cement kg/m^3	Sand kg/m^3	Gravel (3/8) kg/m^3	Gravel(8/15) kg/m^3	Water l/m^3	Admixture l/m^3	PG kg/m^3
RC	400	700	148	938	120	8	-
GPC	280	700	148	938	120	5.6	120

3.3 Chloride Ions Permeability Tests

Chloride ions from sea water penetrate concrete by diffusion or by capillary absorption under the effect of different phenomena. Over time, these chlorides diffuse in the pore solution of concrete and reach the reinforcement bar. To avoid that the chloride ions reach too fast the steel reinforcement, it is essential to choose cement that is resistant to chlorides and ensure a concrete mix with low permeability. In all these issues, the mechanisms of transport of aggressive agents should be considered [8].

The rapid chlorides ions permeability test (RCPT) is performed after curing for 90 days according to ASTM C 1202. This test gives an indication of the penetrability of the chloride ions through concrete under a differential potential of 60 Volts.

The charge Q = It (Coulomb) (1)

The resistivity $\rho = 2\pi e \frac{u}{i}$ (Ω cm) (2)

Where:

e: Thickness of specimen

U: Potentiel

Conductivity $\sigma = \frac{1}{\rho}$ (siemens)

3.4 Gas Permeability Tests

The gas permeability is a transfer property that is commonly used to characterize the durability of the concrete. Thus, the measurement of the oxygen permeability is a reliable measure, relatively simple and usable for the classification of concrete or for an assessment of their potential durability. From this point of view, it can be a good indicator of durability.

The test consists in subjecting the specimen to a constant gas pressure. A schematic representation of the apparatus used is given in figure 5.

The apparent permeability Ka in m^2, is then determined from the measurement of the gas flow Q (in m^3.s^{-1}), in a steady regime with the hypothesis of a laminar flow at a given pressure, using the following formula:

$$K_a = \frac{2\ Q\ L\ P_{atm}\ \mu}{A\ (Pe^2 - P_{atm^2}}\ \text{en}\ (10 - 16\text{m}2) \quad (3)$$

Where:

L (m) is the thickness of the specimen subjected to the flow;

A (m^2) the area of the specimen subjected to the flow.

μ (Pa.s) is the dynamic viscosity of the gaz.

Patm (Pa) is the atmospheric pressure

P (Pa) is the applied pressure.

The intrinsic permeability is calculated by a linear regression according to the Klinkenberg approach.

$$K = K_{int}\left(1 + \frac{\beta}{P_{moy}}\right) \quad (4)$$

$$\text{Avec}\ P_{moy} = \frac{P_0 + P_{atm}}{2} \quad (5)$$

K_{int} is defined as teh intrinsic permeability, P0 the atmospheric pressure, β Klinkenberg constant and β K_{int} is the slope of the Klinkenberg line.

By extrapolation, the value of K_{int} is deduced for a pressure of infinite flow that is when $(1/P_{mean}) \rightarrow 0$. It is the value of the intercept at the origin of the line linking the permeability and the inverse of P_{mean}.

4. Results and Discussion

4.1 Surface Formations

Figure 6 shows an SEM picture of the surfaces of cement pastes specimens conserved in tap water. The microscopic observations showed the presence of a protective layer of calcite in the form of plate or chopsticks on the surface for cement pastes with and without addition of powdered glass. The origin of this layer is due to the presence of carbonate ions which combine with the calcium ions, resulting mostly from the dissolution of the portlandite and also directly from the mineralized water solution.

Figure 7 shows the SEM picture of the surface formations for cement paste specimens conserved in sulfate water. The degraded surface reveals the presence of CSH in the form of honeycomb and few gypsum needles. The presence of the calcite crystals of obtuse rhombohedral form on cement with glass powder paste specimens is clearly observed. In this case, the superficial formation appears denser and tends to make the skin impermeable.

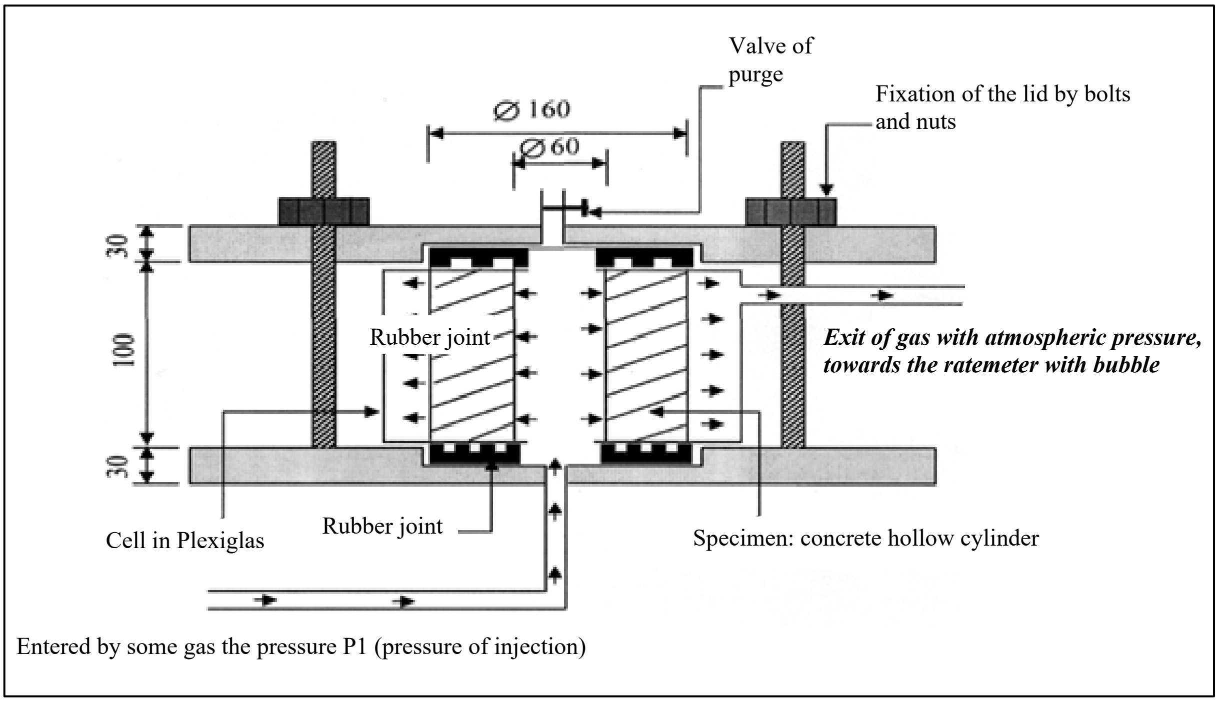

Fig. 5 Schematic representation of the permeability apparatus.

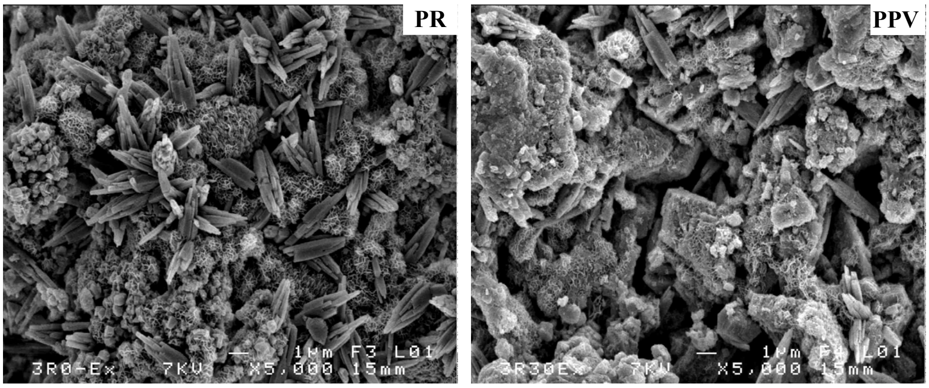

Fig. 6 MEB observation of the external surface of specimens after three months of conservation in tap water.

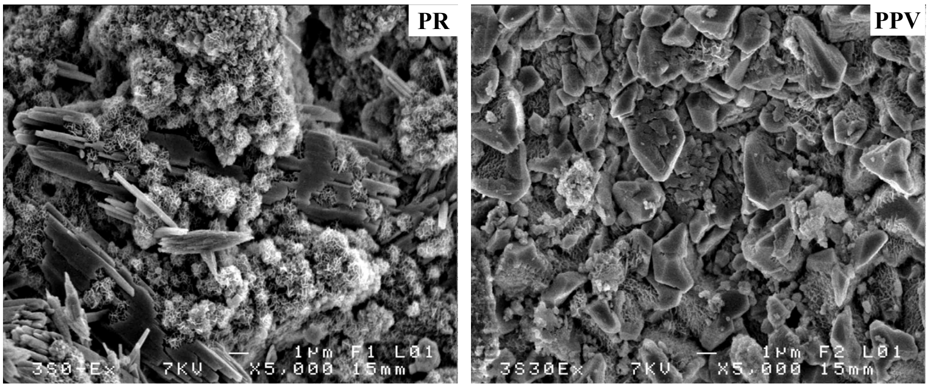

Fig. 7 MEB observation of the external surface of specimens after three months of conservation in sulfate water.

4.2 Internal Microstructure

The microstructure of the hydrates formed in the paste after 3 months is given in figure 8.

Regardless of the environment of conservation, a relatively improved past microstructure with glass powder was observed with relatively more denser interfacial zones that are rich in C-S-H that characterize a high strength concrete with cementitious additions [10]. However, it can distinguish a few lime plates. The presence of ettringite needles is only visible in cavities but the matrix is very dense, it does not promote the development of this phase. The cement paste with glass powder is characterized by the presence of very solid hydrated compounds at different ages, which explains the high impermeability of concrete noticed in what follows.

As pointed out by Scherer [11], concrete damage is frequently attributed to the formation of new (often expansive) solid phases in the porous structure of the material. Under the effect of internal pressures, concrete undergoes high deformations that often lead to its cracking and the failure of the structure. As such, the phenomena of chemical deterioration are in many ways similar to the mechanisms of degradation of concrete under frost attack that result from the formation of ice in the pores of the material. However, chemical weathering phenomena tend to be more complex given that the nature of the formed phases varies depending on the existing conditions [12].

4.3 X-Ray analysis

X-ray diffraction analyses (Figs. 9 to 12) illustrate the influence of the environment of conservation on different formations. The formed calcium hydro-silicates (usually called CSH) are semi-crystalline or amorphous and difficult to identify by X-ray diffraction. Only few rays appear but they are superimposed on the lines of the other crystalline phases.

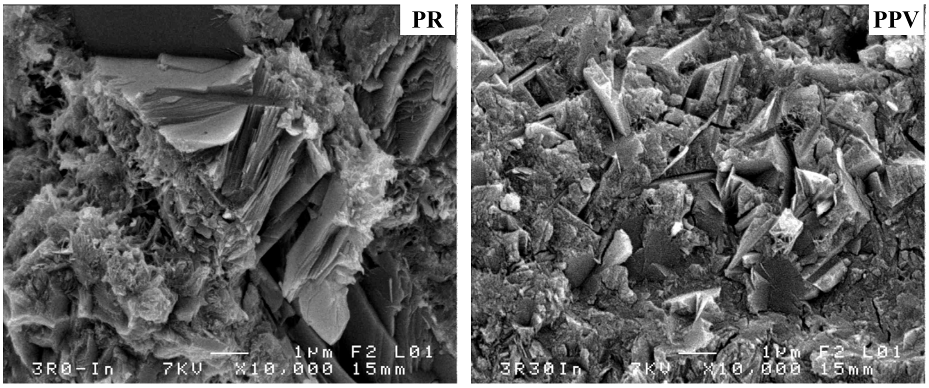

Fig. 8 MEB observation of the internal microstructure of specimens.

Crystalline phases at the inner part of different cement pastes specimen with glass powder appear to be identical regardless of their environment of conservation. The hydration of their anhydrous compounds is stopped, unlike those of the reference paste. This is due to the densification of the matrix.

The calcium hydro-aluminates are crystallized in the form of C_4AH_{13} ($4CaO.Al_2O_3.13H_2O$) for all pastes and are detected by certain diffraction lines. We note the absence of gypsum diffraction lines as it is totally consumed during hydration and contributes to the formation of ettringite. The ettringite and portlandite are visible on all diagrams of cement pastes, even after 3 months of storage in various environments. However, their peaks decrease significantly in the paste with glass powders.

In this study, in addition to a characterization of the available phases, the fineness of the glass powder allows a first semi-quantitative assessment of the development of the hydrated and anhydrous phases as well as the amorphous phases by comparison of the surfaces of the characteristic hump of amorphous phases on XRD diagrams. Regardless of the environment of conservation, the area of the hump for concrete with glass powder is significantly higher compared to that of reference concrete. The reaction of finely ground glass and lime led to the formation of a C-S-H perfectly well defined by its spectrum, whose most important hump, is located at 3.05 Ǻ [13].

4.3.1 Conservation in tap Water

portlandite is in lower quantity in the cement paste with glass powder when conserved in tap water. One part of it has probably reacted with the glass and this explains the densification of the matrix.

4.5 In Sulfate Water

From the three conservations environments, it is observed that glass powder helps to reduce the amount of portlandite in cement pastes. Hence, the pozzolanic effect is very clear.

4.6 Thermodifferentiel and Thermoravitmetry Analysis

Thermodifferentiel and thermogravimetric analyses (TDA and TGA) were conducted to complement the microscopic study and x-ray analysis in order to support discussion on the nature of the observed formations. A sample of 200 mg of paste was analyzed following a linear heating from ambient temperature up to 1000 ° C at a rate of 10 ° C/min.

Figures 13 and 14 show the corresponding TDA and TGA curves. There are three separate peaks and/or significant distinct mass losses. They are linked to structural changes produced by complex physico-chemical transformations in the paste during heating.

- A first mass loss around 20 to 130 °C is due to the evaporation of water and dehydration of the cement paste (C-S-H, ettringite and hydrated calcium mono-carbonaluminate). This dehydration is characterized by a departure of free and bound water [13].
- A second mass loss between 450 to 500 °C corresponds to the dehydroxylation of the Portlandite.
- A third large mass loss, around 670 to 700 °C,

corresponds to the second phase of the C-S-H dehydration, as well as the formation of β C_2S [14].

• Beyond 700 °C, the fourth well accentuated peak corresponds to the decarbonisation of calcite. However, the substitution of 30% of cement with glass powder is well illustrated by a low peak (Fig. 13).

• Finally, an exothermic peak (about 830 °C) is presented for pure paste which is poorly marked for paste with glass powder; it probably corresponds to an activated transformation.

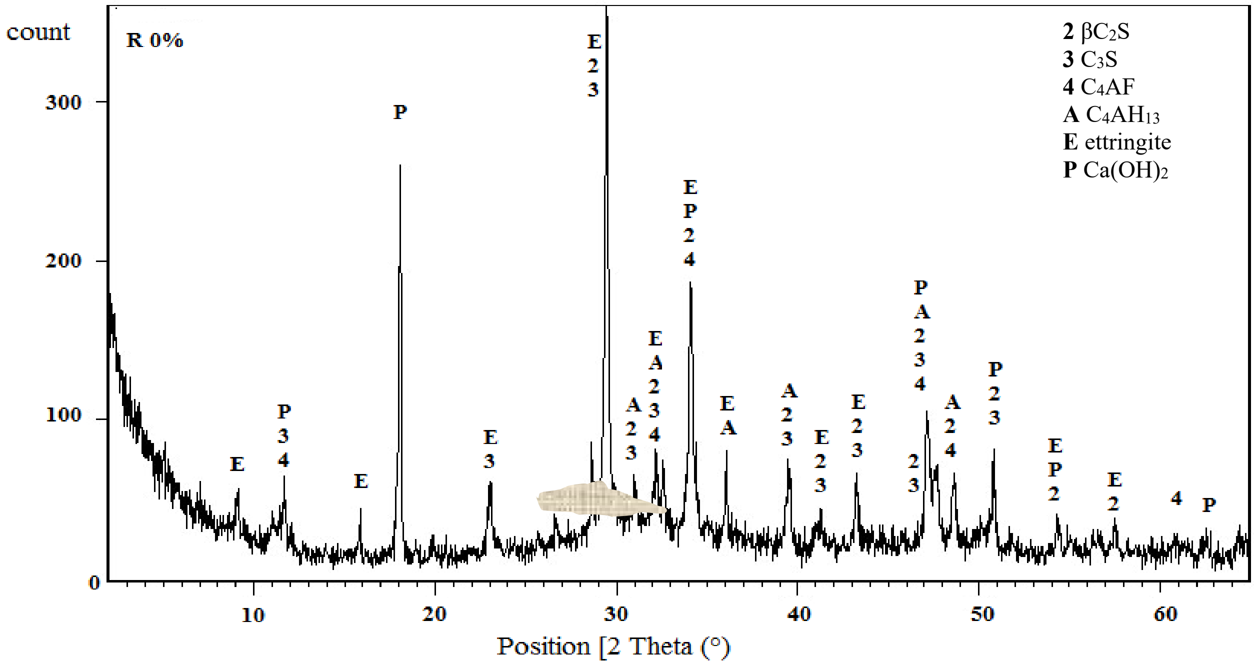

Fig. 9 X-Ray diffraction of cement paste without glass powder after 90 days of curing in tap water.

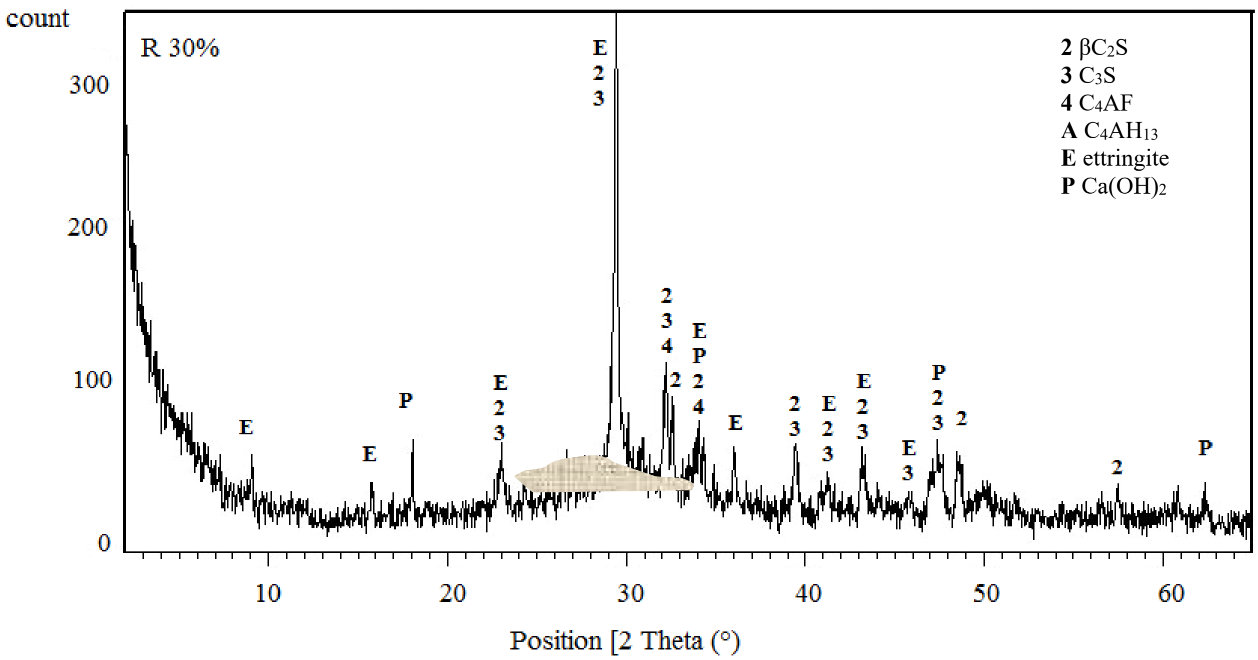

Fig. 10 X-Ray diffraction of cement paste with glass powder after 90 days of curing in tap water.

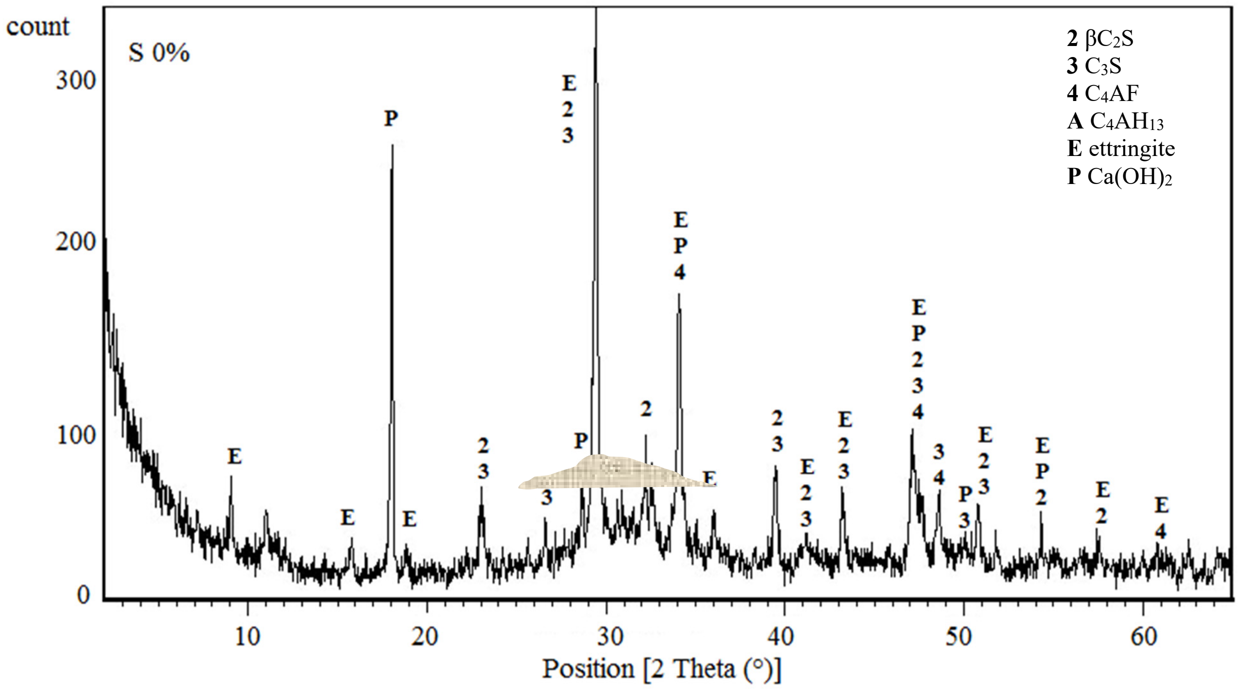

Fig. 11 X-Ray diffraction of cement paste without glass powder after 90 days of curing in sulfate water.

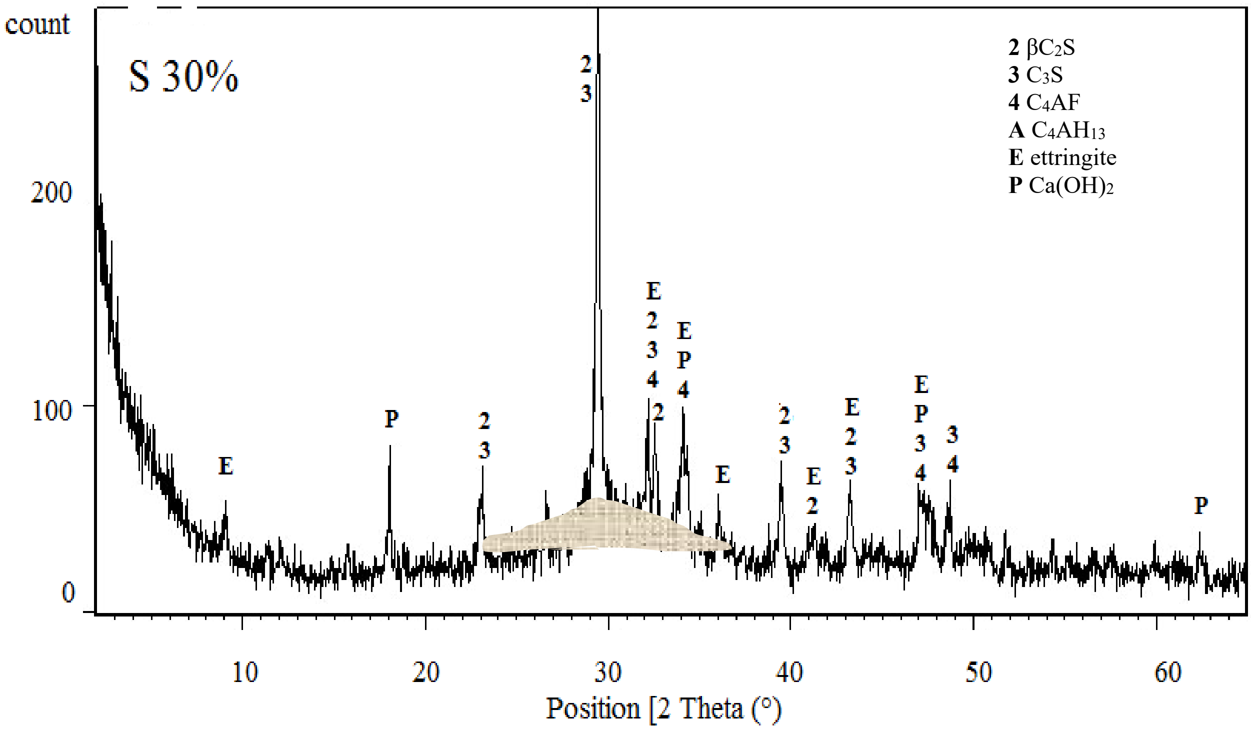

Fig. 12 X-Ray diffraction of cement paste with glass powder after 90 days of curin in sulfate water.

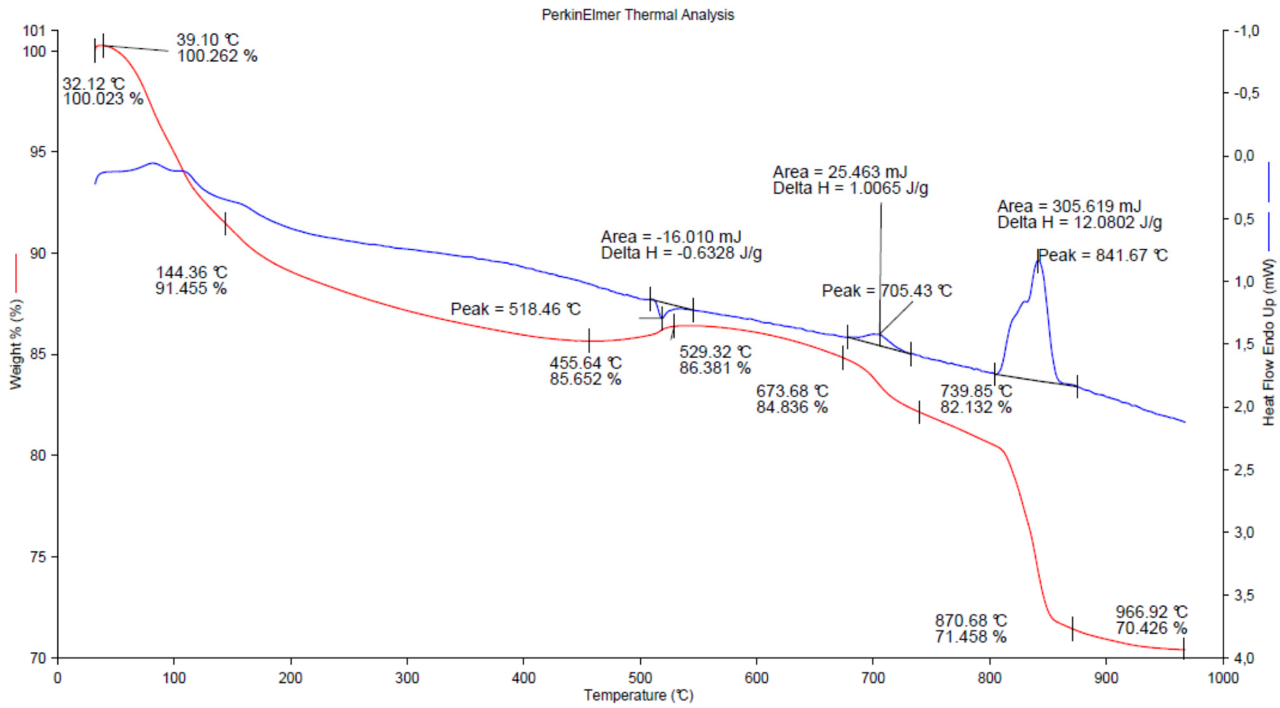

Fig. 13 Thermograph of cement pastes without addition.

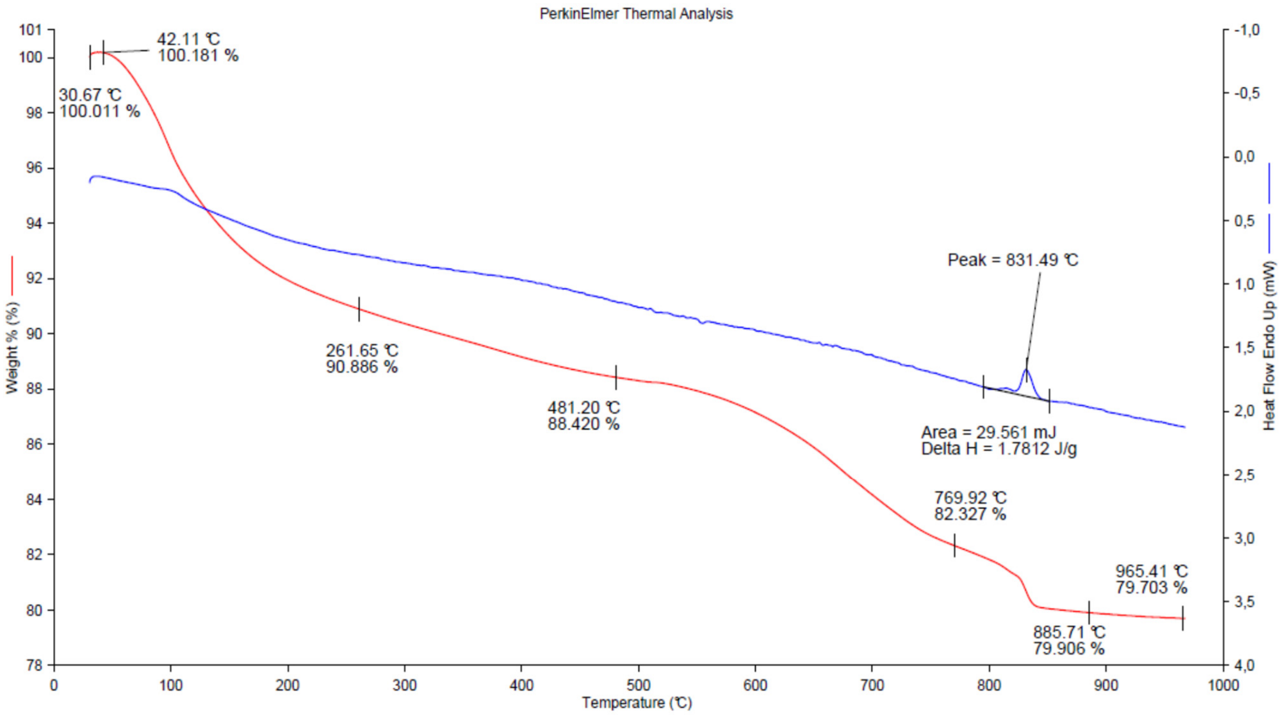

Fig. 14 Thermograph of cement pastes with glass powder.

One should note that the tehrmogravimetyric analysis does not really distinguish the decomposition of the ettringite and that of the C-S-H because they both decompose at about the same temperature. The mass loss related to the dehydroxylation of the portlandite is insignificant with the cement paste containing glass. As reported previously, there is insignificant amount of portlandite.

4.7 Chloride Ions Permeability

The results of the chloride ions permeability tests are summarized in table 7. The variation of current intensity and variation of the charge for concrete cured for 90 days under different environments are presented respectively in Figures 15 and 16. Referring to table 8, which gives a qualitative classification of the penetration of chloride ions through concrete; penetration is very low on concrete with glass powder; However, it is qualified as medium for reference concrete.

The obtained results depend both on the microstructure of concrete as well as the conductivity of the pore solution [15]. A concrete of the same microstructure can show an increase of the resistance to penetration of chloride ions if the conductivity of the interstitial solution is high [16, 17]. However, figure 19 shows that concrete with glass powder presents a very low chloride ions penetrability compared to reference concrete. This confirms that glass powder acts favorably on the microstructure and subsequently on the chemical resistance of concrete.

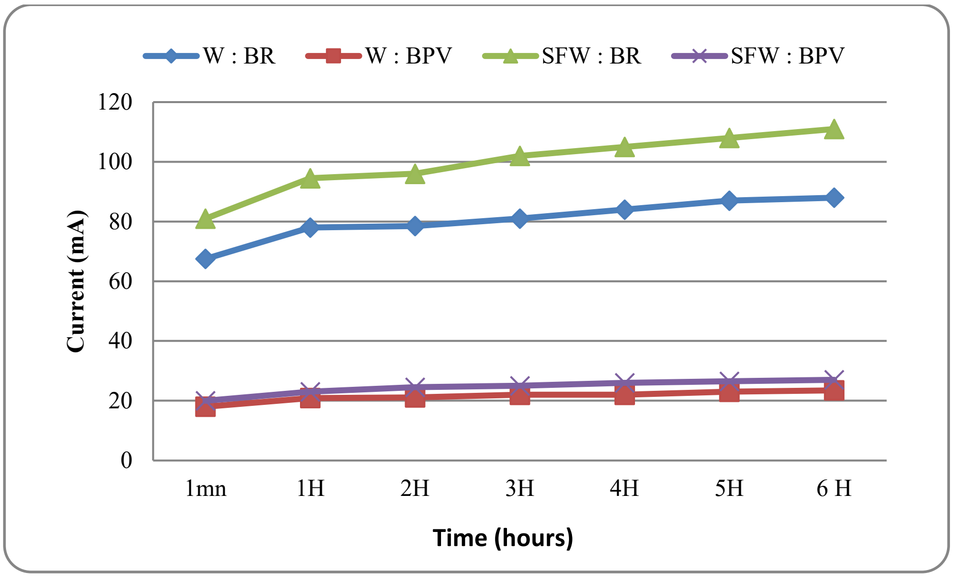

Fig. 15 Variation of current intensity across concrete cured for 90 days under different environments.

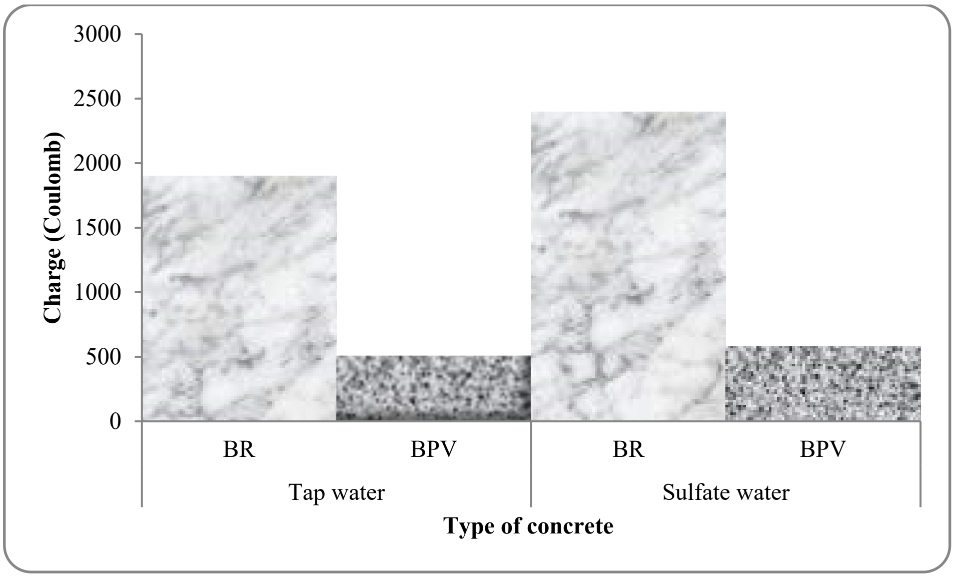

Fig. 16 Variation of the charge for different types of concrete under different curing environments.

Table 7 Chloride ion permeabilty test results.

Environment	Type of concrete	I (mA)							Q (coulomb)
		1mn	1H	2H	3H	4H	5H	6 H	6 h
1	BR	67.5	78	78.5	81	84	87	88	1900.8
	BPV	18	20.9	21.1	22	22	23	23.5	507.6
2	BR	81	90	96	99	105	108	110	2376
	BPV	19	22	22,5	23	24	24	25	540
3	BR	81	94.5	96	102	105	108	111	2397.6
	BPV	20	23	24.5	25	26	26.5	27	583.20

Table 8 Chloride ion penetration.

Passed charge (coulombs)	> 4000	2000 - 4000	1000 - 2000	100 - 1000	< 100
Chloride ion penetration	High	Medium	Low	Very low	negligible

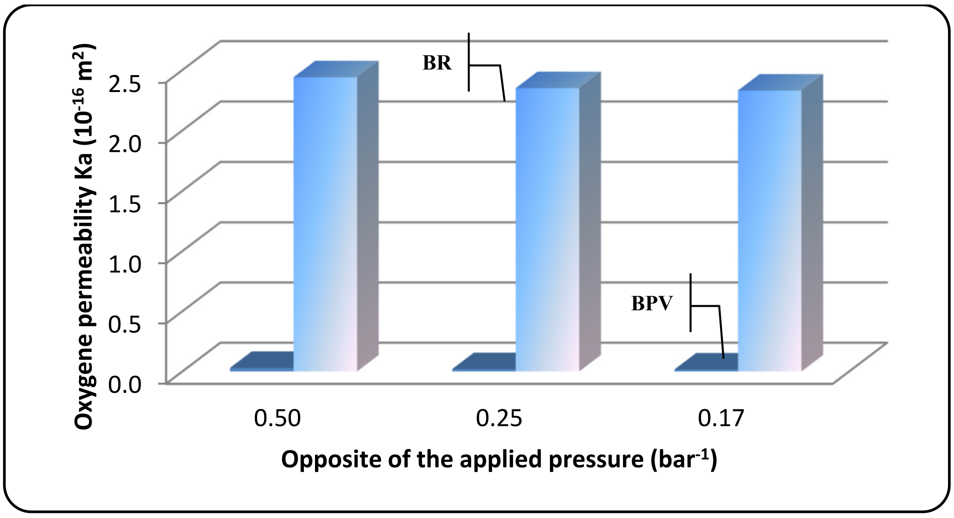

Fig. 17 Variation of the oxygene permeability of concrete.

4.8 Gaz Permeability

The gas permeability, the material property governing the transfer according to Darcy's law, is an indicator of durability. Permeability depends on both the porosity as well as the geometry of the microstructure [9]. Figure 17 shows that regardless of the pressure, the addition of glass powder in concrete strongly reduces the porosity. The addition of glass powder results in the disconnection of existing pores forming an impermeable medium and limiting the transfer of fluids inside concrete.

5. Conclusion

Concrete with SCMS have gained an increased interest in sustainable development of new cementitious materials that are more respectful of the environment. In our study, the reuse of glass waste at the rate of 30% by mass of cement improved the microstructure and reduced the transfer properties. The results of this study have also highlighted that the environment of conservation has no marked effect on hydration. Indeed, apart from their morphologies, the same hydrated compounds are observed in different concretes for the three curing environments.

The oxygen permeability and chloride ions diffusion showed that concrete with or without glass powder concretes have very different behaviors. The diffusion of chloride ions decreases significantly when glass powder is added because of the effect of glass powder on the microstructure which becomes denser and hence resistant to transport phenomenon. The oxygen permeability of mixes with glass powder was lower than that of mixes without glass powder for all three curing environments.

The analysis of the results of TGA and TDA shows that the incorporation of glass powder causes a decrease in mass loss for the dehydroxylation phase of the Portlandite: the quantity of portlandite is lower as it probably reacts by Pozzolanic effect with glass powder. The contribution of glass powder to the binding activity of cement is due to two effects: a physico-chemical and microstructural effect and a chemical effect. On the one hand, it is likely to affect the hydration process of cement as well as the structure of the hydrated products. On the other hand, by its Pozzolanic activity or latent hydraulicity, it reacts in a cementitous medium to form new hydrated products.

From the environmental point of view, the use of glass powder as a substitution to cement could help in reducing the emission of greenhouse gases.

Acknowledgments

The study was performed with the financial support of the French-Algerian research collaboration (CMEP-Tassili) between the laboratory of materials, procedures and environments of the University of Boumerdes and the civil and mechanical engineering laboratory of INSA-Rennes in France. The authors would like to thank EGIDE and the laboratory of chemical science and inorganic materials for their support.

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