EVOLUTION OF DURABILITY AND MECHANICAL PROPERTIES OF ORDINARY PORTLAND CEMENT CONCRETES IN SULPHATES ATTACK

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Abstract:

Concrete in sulphates environments often undergoes significant alterations that often have significant adverse results on its engineering properties. However, the choice of cement type is a very important factor for concrete exposed in aggressive environment. This research presents the effect of two types of cement on the mechanical and microstructure properties of ordinary concretes exposed in aggressive solution dosed with 5% of gypsum ($Ca_2SO_4.2H_2O$). The tests studied in this experimental part were the compressive strength, flexural strength, thermogravimetry, mercury intrusion porosimetry and mass variations of the concrete. The results clearly show that the CEM I 42.5 is suitable for the formulation of concretes exposed to sulphate attack and their properties are better compared with the CEM II/A 42.5.

1 Introduction

The gypsum soils in Algeria occupy about 8.000 Km2, which presents 12.2% of the gypsum soils of the world [1]. Sulfate attack expansion is one of the main factors causing the deterioration of concrete structure mostly at sulfate-rich external environment. Sulphate attack is defined as a reaction between sulphate ions, which can be found in groundwater, seawater, soils, and wastewater [2,3], and hydration products of cement which produce ettringite, causing cracking, expansion, loss of strength and other damages. This reaction is a threat to concrete durability in an aggressive environment. The damage caused by sulfate attack attracted researchers over the years to the search of the degradation mechanism and methods to combat it.

During the design of a concrete structure, one of the most important properties to be considered is durability. To check the durability of cementitious materials, there is an important factor which is the presence of aggressive fluids and their transport [4]. Predictions of durability of the service life can only

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be established on the basis of characterization of properties of the concrete materials and the environment [5,6]. The resisting capacity of concrete material to sulphate attack depends essentially on the quality of its constituents, further the cement matrix. However, the sulphates react with the tricalcium aluminate (C_3A) hydrate of the hardened cement paste resulting in an ettringite which, in the presence of moisture, expands to occupy a greater volume within the concrete.

Previous studies have shown that CEM I 42.5 utilization enhances properties of concrete at longer ages. Khelifa [7] conducted microscopic examination on concrete based on CEMI 42.5 exposed to sulphate attack. He observed that this cement is sulphate resistant and that behaves without evidence of alteration. In addition, Brunetaud and al. [8] reported that concretes made using CEM I 42.5 remained non-sensitive to sulphate attack. This cement confirms that the negative effects of exposure to sulfates result from the interaction between sulfates and phases related to the type of cement. Similar findings were noted by Zaidi and al. [9] that the CEM I 42.5 based concrete present a good permeability performance in an aggressive environment at longer ages. This indicates that the hydrates of this cement fill perfectly the voids in the cementitious matrix. This action prevents the penetration of harmful elements into the concrete. Moreover, results obtained by Khelifa in his experimental study [7], state that the CEMII 42.5 based concrete undergoes degradation and damage in sulphate medium. Moreover, the literature [10-11-12-13] recommend using cement with a low concentration of aluminates or mineral additions. After hydration of cement, the hardened cement paste would result in less tricalcium aluminate hydrate hence the aggressive sulphates have less chance to react harmfully [14].

These data indicate that the sulfate-attack mechanism is complicated, the cracking and expansion of Portland cement concrete under sulfate attack is due to the formation of ettringite and gypsum [15]. Ettringite was defined as the main crystalline phase in the inner zone when gypsum is very close to the surface of concrete [16].

This paper gives a part of study durability properties of ordinary concretes made with CEMI 42.5 and CEMII 42.5 cement exposing to sulphate solution ($CaSO_4$, 2H2O). The investigation was performed by means of compressive strength, flexural strength, thermogravimetry, mercury intrusion porosimetry, and mass variation.

The aim of this research is to possibly substitute the cement used previously, CEMII 42.5, manufactured by the cement plant LAFARGE Algeria (Meftah) with the new cement manufactured by the group GICA (cement plant of Ain Touta), CEMI 42.5, alleged as equivalent to cement sulphate resistant (CRS) in the case of ordinary concretes exhibiting in the sulphate environment. After the study and experimentation, CEMI 42.5 cement gave better results (see results in the article), and it is proposed to substitute CEMII 42.5 cement in sulphate exposures.

2 Experimental investigation

2.1 Materials

In this study, ordinary concrete mixes were prepared using two types of cement, CPA-CEM I 42.5 and CPJ-CEM II 42.5. Chemical composition properties of the cement are shown in Table 1. Natural siliceous was used as fine aggregate, sand with a maximum size of 4 and the coarse aggregate was crushed limestone with a maximum size of 15. The physical properties of aggregates are summarized in Table 2.

In order to achieve proper workability, a superplasticizer was used as a high water reducer polyvalent. It is a new chlorine-based acrylic copolymer generation, dry extract 30% and PH = 4.5-6.5. The superplasticizer dosage was 3.20 Kg/m³ used for all concrete formulations.

2.2 Mixture proportions

The method of concrete formulation was determined by the method Dreux Gorisse, by optimizing the maximum diameter of coarse aggregate [17]. The same grain size distribution was selected for the two types of cement (CEMI 42.5, CEMII 42.5). The two types of concrete were elaborated with a constant W/C ratio equal to 0.48. The mixture proportions are given in Table 3. The characterizations of both concretes in their fresh state are given in Table 4. After 24 hours, one half of the concrete samples were stored in a sulphate environment (5% CaSO4) and the other, in tap water (reference medium) for 365 days at ambient temperature.

3 Test specimens and procedures

Compressive strength test

The compressive loading tests on concretes according to EN 12390-4:2000 [18] were carried out on a compression testing machine of 3000 kN capacity. The specimen used was 150 mm cube. specimens were tested immediately after taking the cubes from curing tank in wet condition. The apparatus used for this research was a TGA Q50 V6.5 Build 196, in the temperature range from 20 to 1,000 °C at a rate of 20°C/min under helium atmosphere.
Mercury Intrusion Porosimetry (MIP)
The testing of porosity and pore structure was performed by Mercury Intrusion Porosimetry (MIP) with an 'AutoPore IV 9500 V1.07' mercury intrusion porosimeter, a 228 MPa mercury porosimeter covering the pore diameter range from approximately 360 to 0.005 µm. This test was carried out on small concrete pieces, weighing approximately 6 g.
Mass variations
The specimens of concrete designed for this test were cubic 150 mm.
Flexural strength test
The flexural strength test on concrete corresponds to EN 12390-5:2000 [19]. The prismatic specimens (70 × 70 × 280 mm) were fabricated and tested in four-point flexure machine of 50 KN capacity.
Thermogravimetry analysis TGA.
To determine the mass of the concrete' s specimens prior to storage, they are weighed right after demolding. The cubes are submerged in both medium and weighed every three months for 365 days. Before weighing, the specimens are cleaned 3 times with distilled water to remove the top layer of concrete. After half an hour, weighing is recorded using a 0.01gr precision scale.
After determination of the concrete' mass of the cubes at all ages, mass variations of immersed specimens were calculated as per following equation:

$$Mass\ Variation(\%) = \frac{m_2 - m_1}{m_1} \cdot 100 \qquad (1)$$

Where m_2 refers to the mass of the specimen at testing time, and m_1 is the mass of the same specimen at the initial age.

Table 1. Chemical compositions of the cements

	Al_2O_3	CaO	SiO_2	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	Cl	LCL	PF	H
CEM I	7.67	64.83	20.94	4.31	1.91	2.18	0.66	0.33	0.04	0.76	4.79	0.6
CEM II	4.99	61.80	18.20	2.78	1.65	2.03	0.68	0.39	0.02	0.92	9.01	0.4

Table 2. Physical properties of fine and coarse aggregates

Properties	Aggregates used				Standard method
	8/15	3/8	0/4	0/1	
Specific gravity (apparent) (g/cm^3)	2.81	2.84	2.76	2.66	P 18-558 P 18-559
Specific gravity (dry) (g/cm^3)	1.52	1.46	1.44	1.46	EN 1097-6
Absorption (%)	1.14	1.47	0.39	0.22	EN 1097-6 P 18-554
Water content (%)	0.00	0.00	1.11	0.30	P 18-554 P 18-555
Porosity (%)	2.83	3.35	-	-	P 18-554
Surface Cleanliness (%)	1.0	1.9	-	-	P 18-591
Kurtosis	8.24	10.03			NF EN 933-3/A1
Los Angeles (%)	20.82	24.48	-	-	NF EN 1097-2 P 18-573
Sand Equivalent (%)	-	-	63.56	74.35	NF EN 933-8
The methylene blue value	-	-	0.33	0.50	NF EN 933-9
Micro-deval (A) (%)	18.2	18.5	-	-	P 18-572 NF EN 1097-1

Table 3. Concrete constituents and mix proportions

Water (L)	Cement content Kg/m^3	Sand 0/1 Kg/m^3	Sand 0/4 Kg/m^3	Gravel 3/8 Kg/m^3	Gravel 8/15 Kg/m^3	Super-plasticizer Kg/m^3
209.97	400	184.37	621.13	279.74	666.88	3.20

Table 4. Concrete properties (*CEMIC: concrete based to CEMI cement, CEMIIC: concrete based to CEMIIC)*

	Slump (cm)	Air Content (%)	Unit Weight (Kg/m^3)
CEMIC	21	2.8	2390
CEMIIC	22.5	3.2	2420

3 Results and discussion

3.1 Compressive and flexural Strengths

The results of the compressive and flexural strength of concrete in both environments were calculated as the mean of the three measurements for all mixtures, and the measured values are presented in Figures 1, 2 respectively. The compressive and flexural strength values and the standard deviation for each individual series of results are shown in Tables 5 and 6 (Each data point is the average for three specimens).

The results were in agreement with the previous studies [8]. While both types of cement are in the same class (42.5), the compressive strength of the concrete made with CEM I was higher than the compressive strength of the concrete made with CEM II.

Compressive strength increases gradually with the increase in the exposure time (until 270 days). This may be attributed to the continuous hydration of the anhydrous cement products, which decreases the volumes of micro-pores leading to a denser structure, which positively influences the mechanical strength, after that the strength starts to decrease.

In the case of the concrete exposed in tap water, high compressive strengths were achieved at the age of 365 days with an average of 67.4 MPa for both concretes. On the other hand, for the concrete exposed in the aggressive environment, the highest level of long-term compressive strength (365days) was achieved for CEMIC (54 MPa) with 34% difference to CEMIIC in sulphate environment.

As shown in Table 6, it can be observed that concretes based on CEMIC exhibited higher flexural strength compared to CEMIIC. From the results, it can be seen that CEMIC and CEMIIC cement concretes showed convergent flexural strengths at early ages, but remarkably divergent at later ages.

The improvement in flexural strength was more obvious at 270 days for CEMI concrete with 27% and 32% difference for both media (a and b respectively). However, at 365 days it can be seen that the strength of concrete decreased for CEMIC levels up to 16%, but is still superior to CEMIIC in sulfate medium with 29% difference. CEMIIC concretes had no change on the flexural strength of the concrete at 270 days and onwards.

Exhibition of the concrete specimens in sulfate medium caused a reduction in 365 day compressive and flexural strengths, in comparison to those of concrete specimens cured with water. However, the results were in the range as defined in the previous studies [20 and 21] where the formation of a sulfated hydrate leads to the creation of micro-cracks, a sign of a greater degradation of the material.

3.2 Thermogravimetry analysis TGA

Figures 3 and 4 shows TGA curves for both CEMIC and CEMIIC in tap and aggressive water respectively aged for 365 days. These curves provide basic information on the thermal behavior of the investigated concretes up to 1,000°C.

In the results of the TGA test, whether in tap or aggressive water, every curve was divided into four zones of interpretation:

The first peak, delimited by 100 and 200°C, for all concrete admixtures, is attributed to the loss of absorbed water by hydrated calcium silicate (C-S-H).

The second zone covers the range between 350 and 450°C, this part concerns just the CEMIC, which shows a relatively complicated behavior in aggressive water, a series of thermogravimetric

bearings that it is difficult to identify. The literature results reported by Xu [22] ascribed these peaks to decomposition of hydrates C_3AH_2 at 250 ° C [23], C_4AH_{12} at 270 ° C [24], and C_3AH_6 at 330 ° C [23]. Around 420° C, a relatively small total mass loss of 1.86% was found for CEMIC in tap water, an almost similar transformation was observed by Sha and al. [25] on cement paste. These authors attribute this peak to the change of crystalline state or the dehydration of a solid solution of Fe_2O_3.

The third zone, which ranges from 450 to 580°C, is explained by the dehydration of calcium hydroxide $CaOH_2$. The difference between mass loss in this stage appears with the exposition medium. Figure 3 showed just two important peaks for CEMIIC, at variance to Figure 4 which shows a suite of endothermic peaks for both concretes. This behavior may be attributable to the sulphate attack effect.

The last endotherm of around 700°C, detected in all concrete admixtures, indicates the decarbonation of calcium carbonate in the hydrated compound.

The TGA curve in aggressive water CEMIC is associated with a total mass loss of 34.68% which is more than that of CEMII 30.48%, but this is not the case for CEMI and CEMII in the tap water, which presented a total mass loss of 30.85% and 34.39% respectively. These basic TGA results emphasize important thermal stability for both concrete admixtures for possible high-temperature applications.

Table 5. Compressive strengths values of concrete at all tested ages

	compressive strength							SD
days	2	7	28	90	180	270	365	
CEMII-TW	28.2	44.9	48.5	57.6	61.9	65.1	66.8	13.76922
CEMI-TW	30.8	42.2	54.0	61.1	62.3	66.2	68.0	13.75619
CEMII-AW	25.5	39.6	41.9	45.5	48.0	58.4	35.8	10.27619
CEMI-AW	26.2	43.7	43.7	50.1	53.0	60.0	54.0	10.93631

Table 6. Flexural strengths values of concrete at all tested ages

	flexural strength							SD
days	2	7	28	90	180	270	365	
CEMII-TW	6.80	7.52	7.98	8.01	8.32	8.30	8.80	0.64458
CEMI-TW	6.15	6.10	5.93	7.59	9.55	13.08	9.58	1.40291
CEMII-AW	5.20	6.17	6.85	8.35	8.93	8.36	8.41	2.6341
CEMI-AW	5.60	6.13	6.00	9.03	9.45	14	11.80	3.20677

TW: tap water; AW : aggressive water ; SD : Standard Deviation

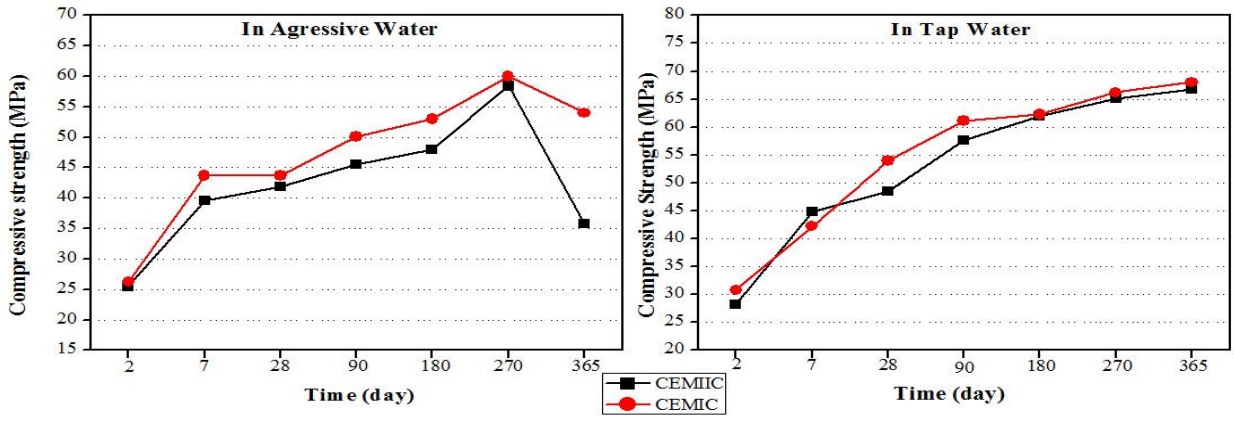

Figure 1. Evolution of compressive strengths

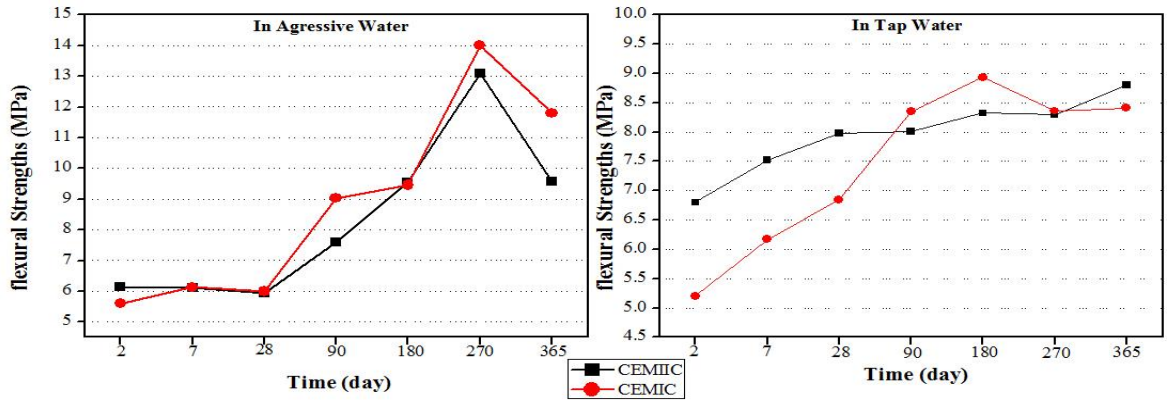

Figure 2. Evolution of flexural strengths

3.2 Mercury Intrusion Porosimetry (MIP)

Mercury intrusion porosimetry (MIP) analysis is widely used to evaluate total porosity and size distributions of pores in concretes. Researchers [26] classified the pores from 10 to 0.05 µm as large capillary pores, from 0.05 to 0.01 µm as medium capillary pores and <0.01 µm as gel pores.

Total porosity and porous distribution of concrete samples at 365 days are presented in figures 5 and 6 respectively.

Figure 5 shows the relationship between the cumulative pore volume and pore diameter in the range of 0.01 to 1000 µm. As can be seen, the lowest total porosity and the smallest diameter pore size, followed by CEMIC. The increase in capillary pore volume in CEMIIC can be attributed to a general microcracking of the cement matrix. The decrease in pore volume around 0.1 µm, which corresponds to the porosity of C-S-H, could mean that ettringite occupies a significant part of this porosity during sulphate attack [7].

It can also be clearly seen that there is not a difference in the total porosity for both concretes in tap water.

In figure 6, CEMIIC showed a higher volume of medium capillary pores and gel pores than CEMIC. The CEMIC pore size distributions presented may be associated with a sound material or at least slightly affected by the sulphate attack. Conversely to CEMIIC which presented peaks clearly more marked, the capillary pores are partially and completely filled with water and reduce as hydration continues. Capillary pores affect the strength and durability of concrete [27]. However, replacing CEM I with CEM II does translate into a shift to smaller pores.

It can be conclude that with the CEMIC, the total volume of the porosity and the refined pore size of concretes decrease, and the most probable pore diameters of concretes shift to smaller pores and fall in the range of less-harmful pore, which indicates that the use of CEMI 42.5 refines the pore structure of concretes in aggressive medium. MIP measurements confirmed the compressive and flexural strengths observations.

3.4 Mass variations

Figures 7 presents the results of the mass variations of different concrete cubes submerged in tap water and sulphate solution.

As shown, the mass of specimens submerged in the tap water increased gradually over time which probably corresponds to the hydration of the cement. Also, it can be seen that the specimens stored in sulphate solution showed much lower loss of mass. This increase of loss is due to the sulphate attack, which is formed as a result of the reaction between portlandite and calcium sulphate (CaSO4). The mass loss of concretes significantly increased with time when exposed to sulphate penetration [28].

Also, as it is illustrated in figure 7, mass reduction in the CEMIC was less than that of the CEMIIIC, these results confirm that the use of CEM I 42.5 cement prevents the formation of deleterious sulfate-related hydrated products [8].

The degradation of cementitious materials under these environmental conditions is represented by the total passage of solution of portlandite and by the progressive decalcification of HSCs, and in other words, of ettringite and monosulphoaluminate [29].

Figure 8 shows a visual inspection of CEMIC and CEMIIC 150 mm cube specimens submerged in sulphate solution after 365 days. This figure shows that apart from the white spots due to the deposition of CaSO4 on the surface of the specimens which are observed in the CEMIIC more than the CEMIC, no macroscopic indicator can detect a sulphate activity within the samples studied.

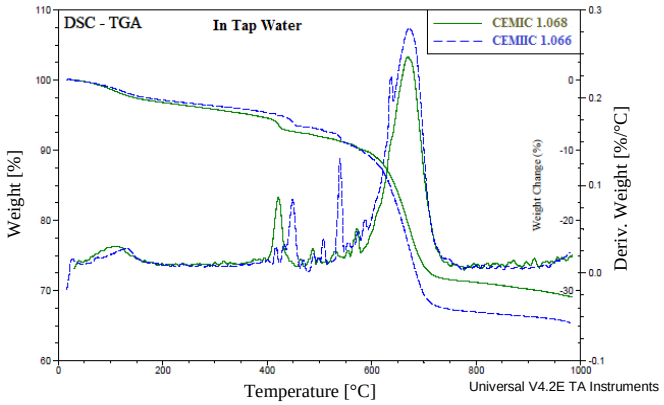

Figure 3.Thermogravimetric analysis output plot for samples in tap water

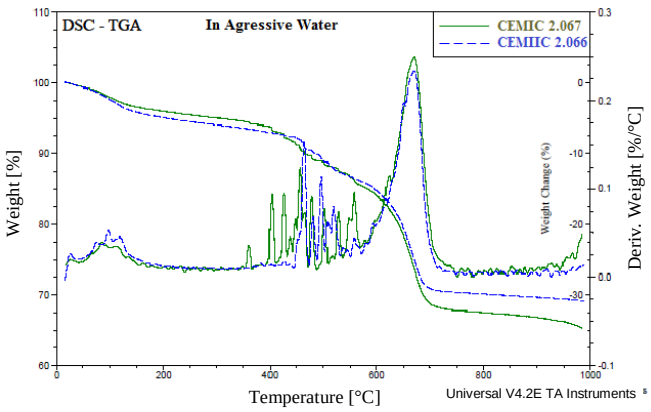

Figure 4. Thermogravimetric analysis output plot for samples in aggressive water

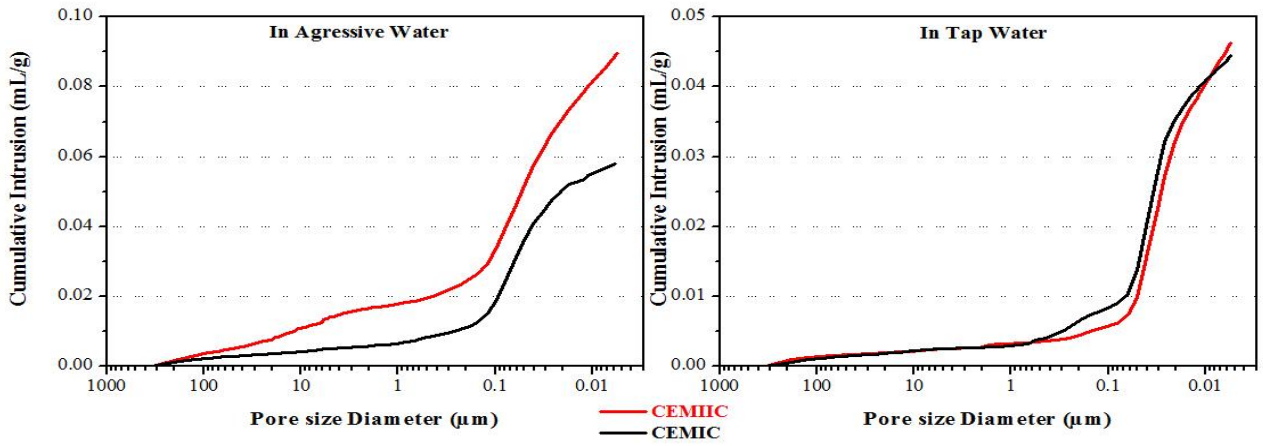

Figure 5. Total porosity of concretes in both medium at 365 days

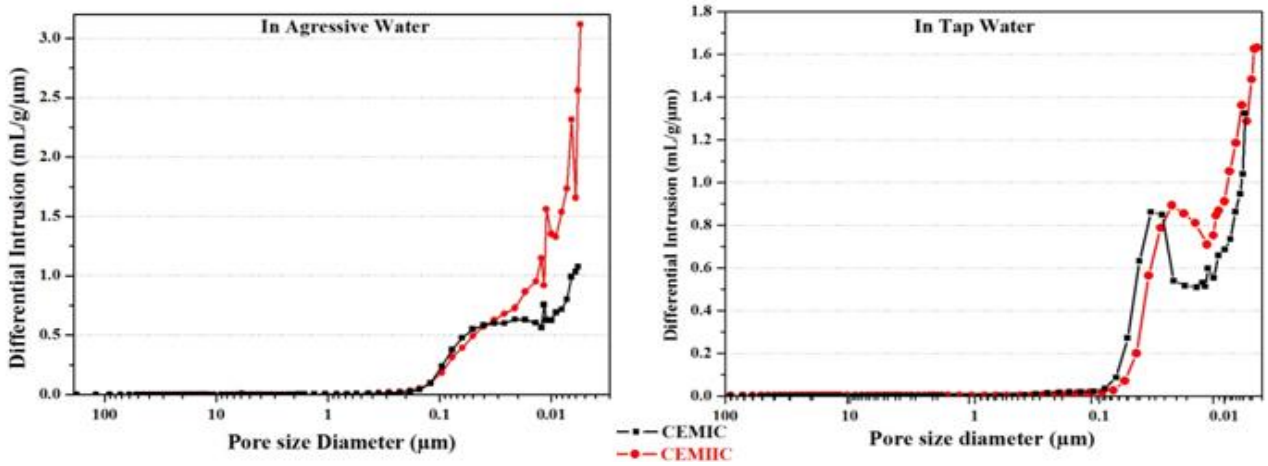

Figure 6. Pore size distribution of concretes in both medium at 365 days

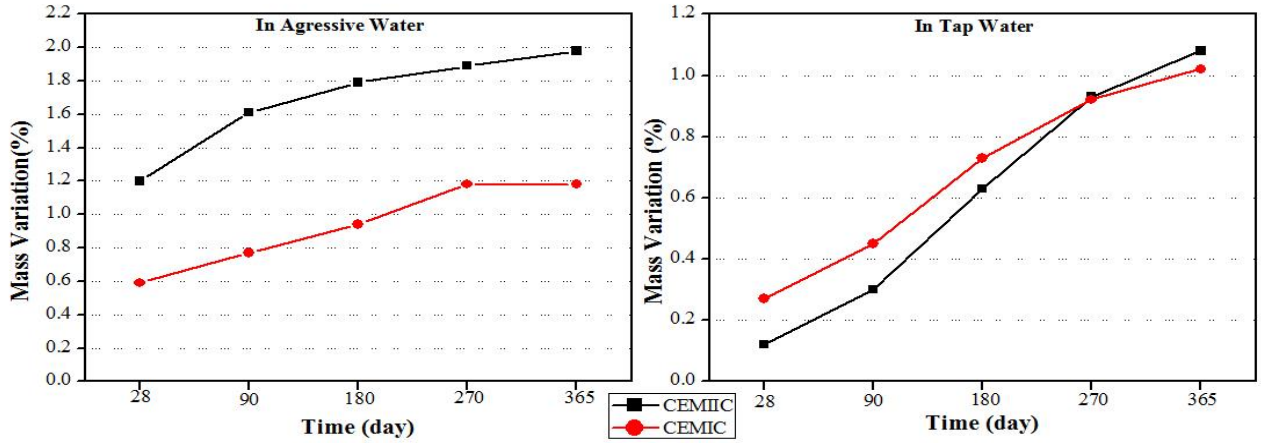

Figure 7. Mass variations of concretes in both medium at all tested ages

Figure 8. Visual inspection of CEMIC and CEMIIC 150 mm cubes after 365 days

4 Conclusion

The experimental results previously discussed lead to the following conclusions:

1 The behavior of exposed samples in tap water and sulphate environment is different; the referenced medium confirms that the negative effects of sulphate attack results from the interaction between sulfates and the type of cement.

2 The concrete with the lowest resistance to sulfates was that of CEM II 42.5 due to the decrease in compressive and flexural strength at 365 day ; unlike CEMI 42.5 which presented good values of strength.

3 According to the TGA results, both concretes show important thermal stability, which allows them to be used for high-temperature applications.

4 The total pore volume of CEMIIC is higher than CEMIC. This is probably attributed to a general microcracking of the cement matrix. CEMI is less permeable, which confirms the compressive and flexural strengths observations.

5 CEMIC presented mass reduction less than that of the CEMIIC, which confirm that the use of this CEM I 42.5 cement prevents the formation of deleterious sulfate-related hydrated products.

6 No damage was observed in the specimens exposed to sulfate solution for 365 days.

The various results confirmed that concretes made using CEM I 42.5 remained non-sensitive to sulphate attack.

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