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CHARACTERIZATION OF THE REACTIVITY OF MINERAL ADDITIONS BY DIFFERENT MICROSTRUCTURAL AND MECHANICAL APPROACHES

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Abstract: This paper presents an experimental study aimed at evaluating the reactivity of natural and industrial local mineral additions (pozzolan, slag and limestone) by different microstructural and mechanical approaches. Binary, ternary and quaternary cement compositions were prepared with partial replacement of the clinker by additions limited to 20%, according to CEM II / A cement specifications.

The reactivity during the hydration process is characterized by the hydraulic power of the additions to react with the water and the hydrates of the cement and the pozzolanic capability of fixing the portlandite to form new mineral phases which contribute to the resistance as much as the hydrated products of cement.

An experimental methodology was established for the reactivity illustration of the additions by a microstructural approach based on a study of the physicochemical and microstructural properties realized by X-ray diffraction (XRD), the scanning electron microscopy (SEM) on pastes. Further, thermogravimetric analysis (TGA), mercury microporosity (MIP) on mortars at 28 days of age was performed. A mechanical approach based on compressive strength at 2, 7 and 28 days to determine the activity index according to ASTM C 618 has been undertaken.

The results obtained show a good correlation between the microporosity, X-ray diffraction and the resistance activity index, in particular for the slag, as for the thermogravimetric analysis that clearly confirms the pozzolanic activity of the pozzolan addition.

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As for mechanical performance, a binary mortar with 20% slag showed a better compressive strength at 28 days (49.40 MPa) with a reactivity index (87.90%) compared to the control mortar. Further, the slag developed better resistance (49.53 MPa) in combination with the other additions for quaternary mortars (pozzolan, limestone and slag) with an optimal rate of 5, 5 and 10%, respectively. Nevertheless, the pozzolan showed better compressive strength values compared to the slag for the binary mortars at young age 2 and 7 days of 25.67 and 32.07 MPa, respectively.

Keywords: *reactivity, pozzolanicity, mineral additions, physico-chemical properties, microstructure*

1. INTRODUCTION

With the new concept of sustainable development, that is based on social, economical and environmental dimensions. The industrial world still a principal origin of carbon dioxide gas emission and the industry of cement is classified in the first rank of CO_2 release in the atmosphere. Some research data revealed that the production of any single tone of cement lead to 0.9 ton of carbon dioxide release in the air (Aïtcin 2001).

For any positive sustainable development contribution, the use of additions either mineral or industrial in the cement production has become a necessity; in regards to the beneficial aspects of such use from the economical functional and environmental side. Thus, the valuation of industrial and abundant natural wastes for the purpose of clinker replacement. The utilization of additions is fundamentally related to the gained technical advantages mainly, its hydraulic reactivity and pozzolanic characteristics. These additions could react in the presence of batching water to produce new hydrated components with binder properties (Venuat 1989).

The reactivity of the additions influence significantly the cement matrix properties and contribute to modify in different ways on the formed hydrates within complex reactions mechanisms. Many researches agree on the tendency to resume the main effects of additions on the cement matrix. The granular effect, the physico-chemical effect acting on the rheological side and thus on the microstructure of the cement matrix at the fresh hard and state (Nonat 1994; Baron 1996; Uchikawa and Hanehara 1996; Mounanga 2003; Lawrence et al. 2005).

The effect is related to the chemical activity which appropriate to certain types of additions in the cement media during the hydration process. However, it can be concluded that the pozzolanic activity of mineral additions remain an important research field of interest in order of a better understanding and a further quantitative and qualitative analysis of the use of these additions (Mejía Durán et al. 2006; Itim et al. 2011; Singh and Gupta 2016).

Several studies have been carried out on local additions, that aims to limit the use of high clinker content by replacing it with granulated blast furnace slag in the algerian cement plant factories. On the other hand, they tried to characterize this addition by its degree of reactivity (Naceri and Messaoudene 2006; Behim 2005; Beddar and Clastres 2013). Others presented an experimental study dealing with effect of industrial pozzolana on the mechanical properties and durability of ordinary mortar (Rabehi et al. 2014). In addition, research study has reported the effect of blast furnace slag and natural pozzolan on the rheological and mechanical properties of mortars (Oudjit et al. 2011).

In this context, the present study aims the global characterization of the mineral additions reactivity according to a microstructural approach of experimental methods using DRX and MEB on the binary, ternary and quaternary cement pastes based on these additions. Also, the thermogravimetric (ATG) and mercury micro porosity (MIP) tests are undertaken on the prepared alike mortars. Further, A mechanical approach based on the evaluation of the strengths for the different mortars compositions is done; thus to determine the activity index of such binders in accordance with standard (ASTM C618, 93).

The study herein, the reactivity effect deep analysis of additions such as pozzolana, slag and limestone; the significance of their interactions on the microstructural, physical and mechanical behavior of tested pastes and mortars, once utilized separately or in combined cases is considered in the present research.

2. MATERIALS AND TEST PROCEDURES

2.1. MATERIALS

The materials used in this experimental study are purely local materials, which are available in large sources throughout the Algerian countryside.

- Clinker is the main binder obtained from one of the large cement company in Algeria located at Ain Kebira (Sétif-Algeria), The chemical and mineralogical composition of the clinker is presented in Table 1. This contains a high content of Alite "Alimental Cement", which can better develop the initial resistances. On the other hand, its C_3A content is low, which makes it possible to reduce the release of heat during its hydration process and make it more resistant to aggressive agents (Oudjit et al. 2011).

The X-ray diffraction diagrams presented in Fig. 1, illustrates the mineralogical components of the clinker.

- The gypsum used as a setting regulator is delivered by a deposit quarry at Djemila (Sétif, Algeria), its chemical composition is presented in table1.
- The granulated blast furnace slag is a by-product of the El-Hadjar steel factory from Annaba (City East of Algeria), this is used as an active mineral addition, the slag used in this experiment contains especially the oxides having a considerable content which are (CaO-SiO_2-Al_2O_3-MgO). It consists of more than 2/3 by mass

of the sum of CaO, SiO_2 and MgO, the remainder being Al2O3 with further small amounts of other oxides (Behim 2005). Its quality can be estimated through the chemical composition (Table 1) by its hydraulic coefficient "M_b" as a basicity indices. The most commonly used indices are summarized in Table 2.

Table 1. The chemical and mineralogical composition of materials used

Constituents (%) / Materials	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	SO_3	K_2O	CL	Na_2O
Clinker(C)	21.33	4.60	66.39	5.48	0.28	0.71	0.31	0.004	0.35
Gypsum(G)	10.05	2.99	26.90	1.55	3.86	30.33	0.41	0.007	0.05
Slag (S)	29.00	11.30	46.00	1.35	8.12	1.98	0.48	0.047	0.67
Pozzolan (PZ)	40.00	18.80	15.90	9.00	5.23	2.00	2.26	0.012	0.41
Limestone(L)	15.20	2.34	78.70	1.73	1.04	0.56	0.32	0.003	0.15

The mineralogical composition of clinker			
C_3S	C_2S	C_3A	C_4AF
69.37	8.83	2.92	16.68

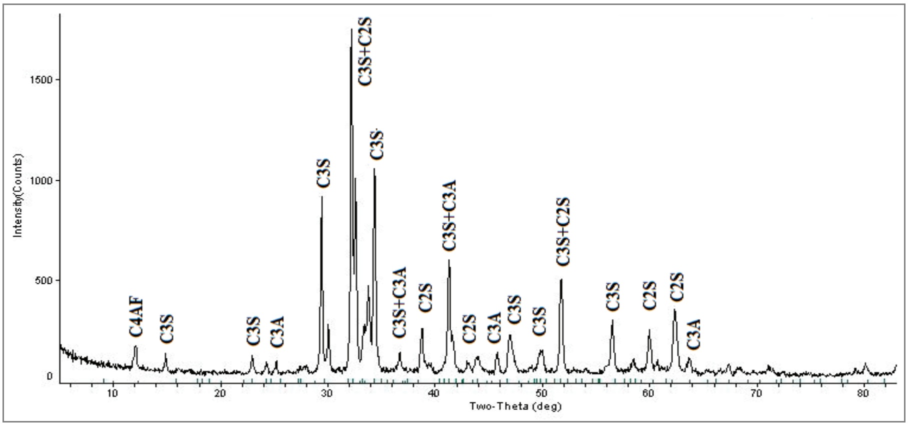

Fig. 1. X-ray diffraction diagrams of Clinker (kαCu radiation)

Table 2. The "M_b" basicity indices for the slag addition (Naceri and Messaoudene 2006)

Basicity modulus (M_{bi})	Value	Comment	
M_{b1} = (CaO +MgO) / SiO_2	1.87	>1	The basicity index (M_{bi} > 1), therefore the slag is basic.
M_{b2} = CaO / SiO_2	1.59	>1	
M_{b3} = (CaO +MgO) / (SiO_2 +Al_2O_3)	1.34	>1	

Figures 2–4 present the microstructure texture of additions slag, pozzolan and limestone, respectively: Images (a–f) show the observed scanning electron microscopy for the composition and distribution of chemical elements (Si, Ca, Fe, Al and Mg) of the additions. Part (g) presents the X-ray diffraction patterns of each addition.

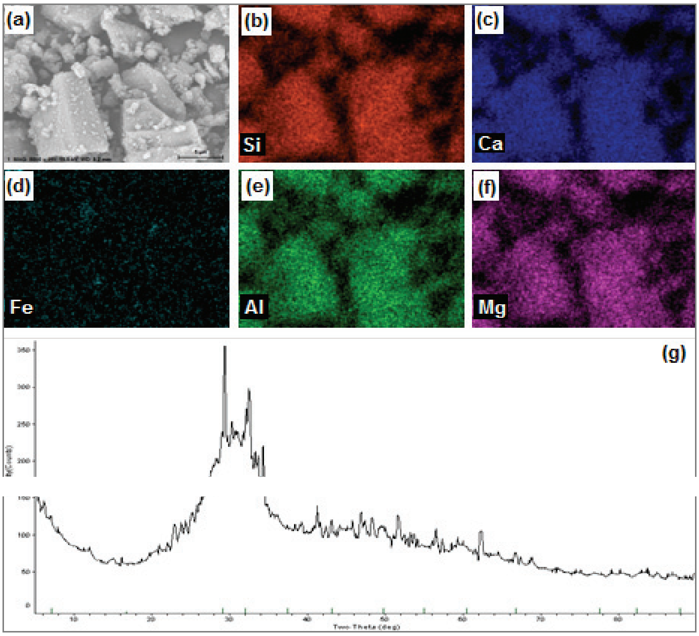

Fig. 2. (a)–(f) – scanning electron microscopy of the slag,
(g) – X-ray diffraction patterns of the slag

- The natural Pozzolan rock extracted from the Bouhamidi deposit located at Béni-Saf west of Algeria, used as an active mineral additive. that natural Pozzolan contains higher levels of silica, alumina and iron oxide and a lower rate of free lime compared to slag addition (Table 1).
- The limestone filers used are obtained from the crushed stone of the Ouled Eddouane deposit (Sétif-Algeria), used as an inert mineral addition, its chemical composition is presented in Table 2.
- The standardized sand is used for mortars preparation according to the European standard (NF EN 933-2, 1996; NF EN 933-1, 1997).

The physical properties of the various materials used (Specific density, Bulk density, Fineness) are given in Table 3. The fineness of the materials used is taken very close by varying from (3200–4000) cm^2/g to fix the effect of the fineness in this study.

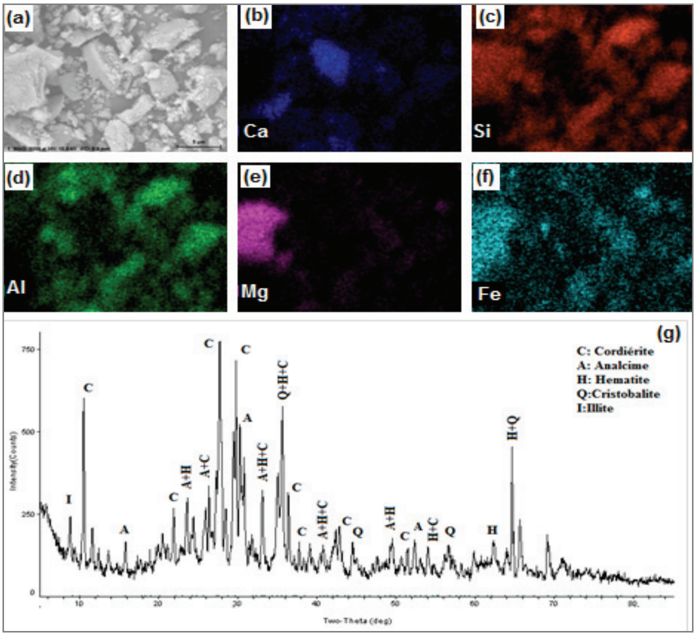

Fig. 3. (a)–(f) – scanning electron microscopy of the pozzolan, (g) – X-ray diffraction patterns of the pozzolan

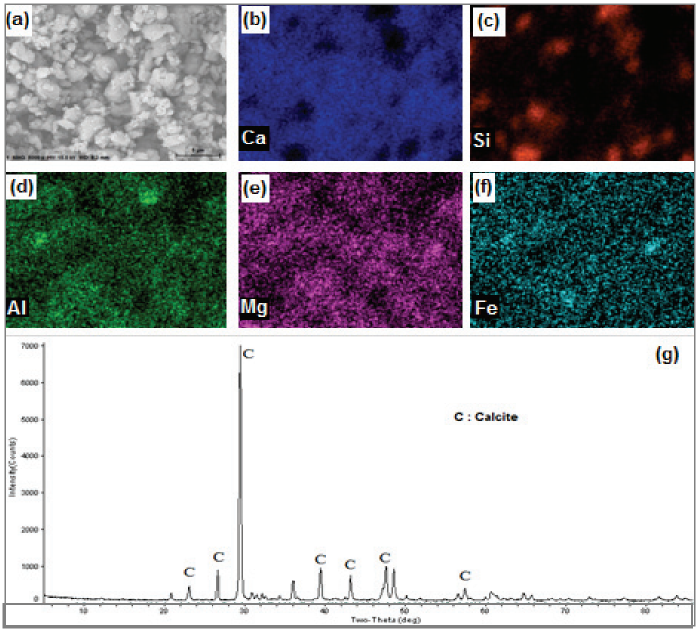

Fig. 4. (a)–(f) – scanning electron microscopy of the the limestone,
(g) – X-ray diffraction patterns of the limestone

Table 3. Physical properties of raw materials used

	Clinker (C)	Gypsum (G)	Pozzolan(PZ)	Limestone(L)	Slag (S)
Specific density (g/cm^3)	3.22	2.50	2.62	2.68	2.79
Bulk density (g/cm^3)	1.04	0.908	0.950	0.972	0.980
Fineness (cm^2/g)	3490	3500	3900	3800	3215

2.2. MIXTURES PREPARATION

The tests were carried out on cement pastes and mortar mixes based on different compositions of cements with a maximum rate of mineral additions of 20%, according to CEM II/A cement specifications for this type of cement; which is the substitution level of the clinker by additions (Pozzolan, Limestone and Slag) in order to make binary, ternary and quaternary cement. The limestone is considered as an inert addition at a limited content of 5%. The compositions and nomenclature of the prepared mixtures are summarized in the Table 4.

Table 4. Composition of cements studied (% by weight of cement)

Mixtures	Clinker(C)	Gypsium(G)	Mineral Additions percentage (%)		
	(%)	(%)	Pozzolan (PZ)	Limestone (L)	Slag (S)
Control	95	5	–	–	–
M1	75	5	20	–	–
M2	75	5	–	–	20
M3	75	5	10	–	10
M4	75	5	10	5	5
M5	75	5	5	5	10

The physical properties of the various cements with additions (Specific density, Bulk density, Fineness) are given in Table 5.

Table 5. Physical properties of cements with mineral additions studied

Mixtures	Specific Density	Bulk density	Fineness
	(g/cm^3)	(g/cm^3)	(cm^2/g)
Control	3.165	1.035	3480
M1	3.063	1.017	3560
M2	3.083	1.023	3426
M3	3.073	1.020	3490
M4	3.071	1.020	3520
M5	3.076	1.021	3475

2.3. METHODS

- The different prepared pastes of cement compositions were analyzed at 28 days by X-ray diffraction, Rigaku DMAX III/C with Cu Kalpha radiation (K = 1.5406 Angstrom) and a pitch size of 1.2°/min in the range of 5–90°. Phase identification was carried out using the Jade software and with the help of files on powder diffraction standards (JCPDS, 1967).
- The pastes of cement studied were observed with scanning electron microscopy (Hitachi S-3400N SEM), to provide surface electrical conductivity, the samples were coated with a thin layer of carbon which was sputtered.
- The test for the thermogravimetric analysis was carried out on mortars, the apparatus used for this work was a Stanton Redcroft TG-760. The objective of this test is to determine the percentage of the total amount of $Ca(OH)_2$ from the thermogravimetric. The curves for this purpose are obtained by plotting the cumulative weight loss with respect to the temperature
- The mercury microporosity (MIP) test was carried out by the apparatus (Micromeritics Autopore IV) on samples of mortars at the age of 28 days, made from various compositions of prepared cements. The purpose of this test is to quantitatively determine the porous structure and to obtain very reliable information concerning the distribution of the volume and the size of the pores.
- The mechanical tests were undertaken on prismatic specimens (4 × 4 × 16 cm) in accordance with European standard (EN 196-1, 2005). The three prisms in a single mold were a cast then kept at in a room at ambient conditions of 20 °C and 50% relative humidity. These were demolded after 24 hours and cured in a gypsum saturated water medium.

3. RESULTS AND DISCUSSION

3.1. X-RAY DIFFRACTION ANALYSIS AND X-RAY FLUORESCENCE CHEMICAL COMPOSITION OF THE CEMENT STUDIED

Table 6. Chemical composition of the cement pastes studied

Mixtures	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	Fe_2O_3	P_2O_5	TiO_2	Na_2O
Control	1.75	3.90	16.54	2.53	0.69	70.15	4.69	0.14	0.15	0.49
M1	2.23	6.04	19.91	2.60	0.84	61.07	5.64	0.21	0.50	0.95
M2	2.68	5.15	18.28	2.67	0.60	65.41	4.55	–	–	0.56
M3	2.44	5.98	19.45	2.57	0.61	61.99	5.68	0.18	0.35	0.76
M4	1.99	3.82	16.10	2.67	0.62	69.11	4.27	–	–	0.38
M5	2.12	4.54	16.92	2.75	0.69	67.76	4.62	–	–	0.60

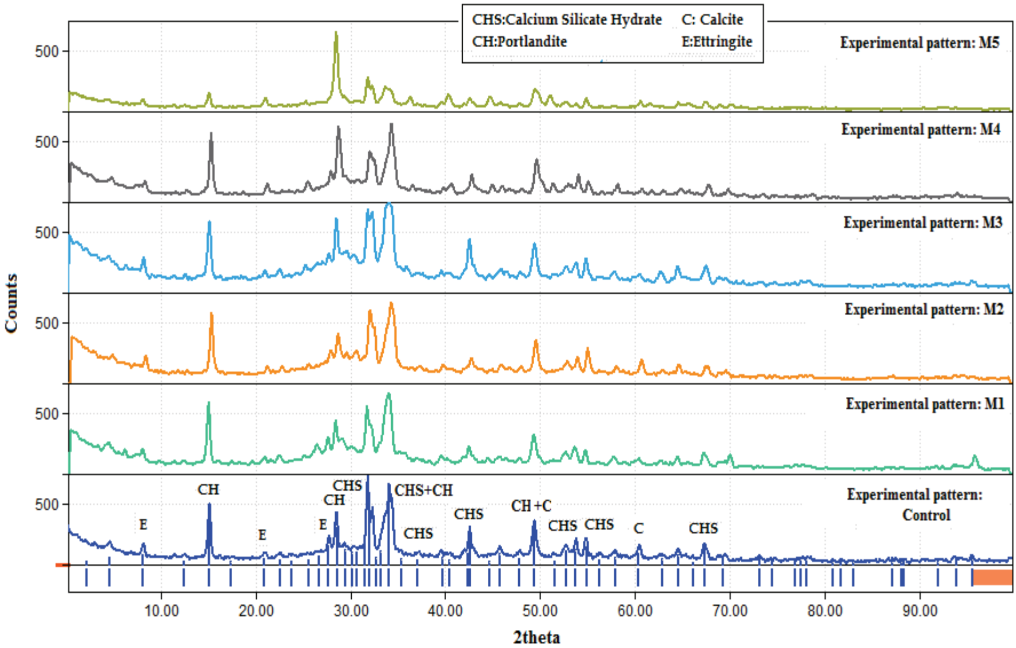

Fig. 5. X-ray diffraction patterns of control and cement Pastes (M1, M2, M3, M4 and M5)

The X-ray diffraction pattern analysis of the studied pastes in figure 5, clearly shows that all these have a crystallized structure composed of mineral phases translated by peaks in the plotted graph.

The qualitative analysis in terms of nature of hydrates shows the presence of calcium silicates hydrate (C-H-S), ettringite (E), portlandite (CH) and calcite (C), pastes with additions develop the same hydrates as the control one.

Quantitative analysis shows that the fundamental hydration product is calcium silicate hydrate C-S-H, which plays a key role in the development of mechanical resistance and durability, the dominance of C-S-H testifies a good reactivity of the additions , the case of binary (M1 and M2) and ternary (M3) pastes prepared with slag and pozzolan compared to control, according to Taylor (Taylor, 1997) the slag hydration products are the same as those of ordinary Portland cement, except that C-H-S formed around grains have a Ca/Si ratio (1.55) lower than that obtained for an ordinary portland (1.7).

On the other hand, a rather weak intensity of hydrated calcium silicate is recorded C-H-S for quaternary pastes (M4 and M5) containing limestone, which does not react chemically to form new products.

In addition, the quantification of the Portlandite (CH) which is manifested with lower frequencies compared to (C-H-S), also makes it possible to estimate the pozzolanic activity. The quaternary cement pastes (M4 and M5) in which limestone has been associated, the portlandite is present with a relatively high intensity compared to the pasta of the ternary cements with active additions (pozzolan and slag).

This shows that the portlandite released from hydration of the clinker is not fixed by the limestone and remains free, so (CH) is more advantageous as long as the pozzolanic reaction is slower than the hydration reaction for cement which can only occur after the hydrolysis of C_3S and C_2S which form calcium hydroxide (Ramezanianpour 1987). Also, ettringite (E) hydration product of C_3A (tricalcium aluminate), attests the reactivity of the additions and it manifests itself with higher intensities especially in the pastes (M1 and M2).

The reactivity of the additions does not modify the hydration of the cement clinker; it completes and integrates the hydration process, because it results in a lower content of portlandite and an increase in calcium silicate hydrates.

3.2. SCANNING ELECTRON MICROSCOPY OBSERVATION OF THE PASTES STUDIED

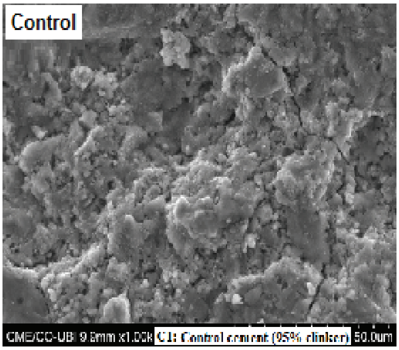

Fig. 6. SEM observation of cement paste confected with control cement (Control)

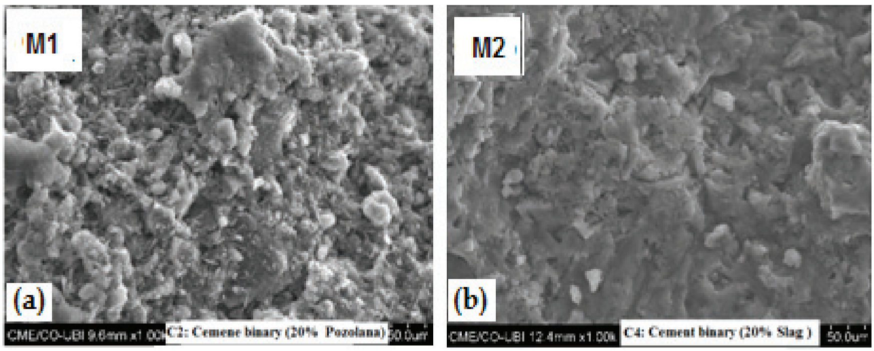

Fig. 7. SEM observation of cement pastes confected with binary cements: (a) pozzolan, (b) slag

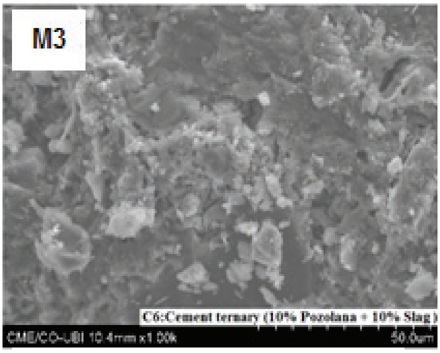

Fig. 8. SEM observation of cement pastes
confected with ternary cement (pozzolan and slag)

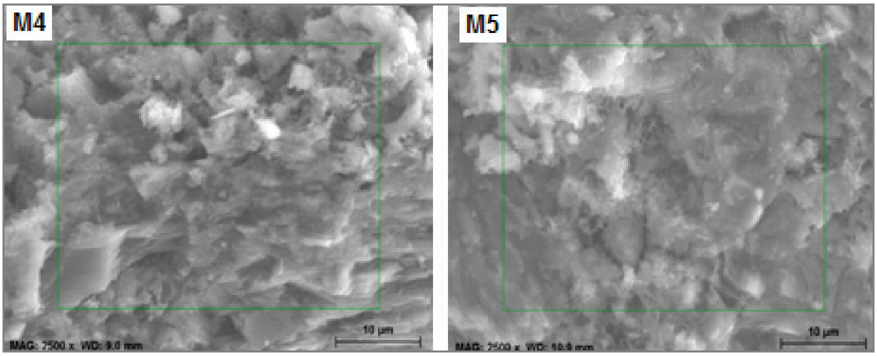

Fig. 9. SEM observation of cement paste confected with quaternary cement
(pozzolan, limestone and salg)

The analysis of the images observed at the SEM shows the distribution of the minerals in the microstructure of the studied pastes and provide information on the nature and morphology of the hydrates formed during the hardening. The presence of a very dense gel of hydrated calcium silicate (CHS), which clearly dominates the microstructure of pastes, C_2S and C_3S lead to produce a CSH gel of about 82% and 61%, consecutively (Abbas 2017). We observe fewer portlandite (CH) plates, especially in ternary pastes with pozzolan and slag compared to quaternary mix with limestone, appearance of a few needles of ettringite (E) especially in quaternary cement, the products of hydration of C3A (tricalcium aluminate) are generally ettringites (E) in the most advanced stage of hydration and monosulfoaluminate in a later stage (Abbas 2017).

As for the microstructural texture and the distribution of minerals, it can be noted that M1 with pozzolan (Fig. 7(a)) has a microstructure with a fairly high microporosity comparable to that of the control (Fig. 6) unlike the mix (M2) with slag (Fig. 7(b)) which shows fewer pores and a denser microstructure.

The porosity, the pore size distribution, and the microstructure are related to the hydrates formation process and their distribution. Therefore, the high porosity of pozzolan is due to the nature and the hydration process that takes all its magnitude at long duration (Amouri 2009). Previous studies have proved the effect of the high specific surface, on the mortars with ultrafine additions (pozzolan) that present porosity higher than mortars with fine additions such as slag (Badreddine 2004).

The quaternary cement with limestone (M4, M5) (Fig. 9), shows a rather dense structure and prove that limestone plays a role of preferential nucleation site during the hydration reactions of the cement. Further, it generates a better distribution of hydrates especially portlandite in hardened state (Caré et al. 2000). This correlates well with the presented results of the microporosity (MIP) (Fig. 11).

3.3. THERMOGRAVIMETRIC ANALYSIS (TGA) AND $Ca(OH)_2$ QUANTIFICATION

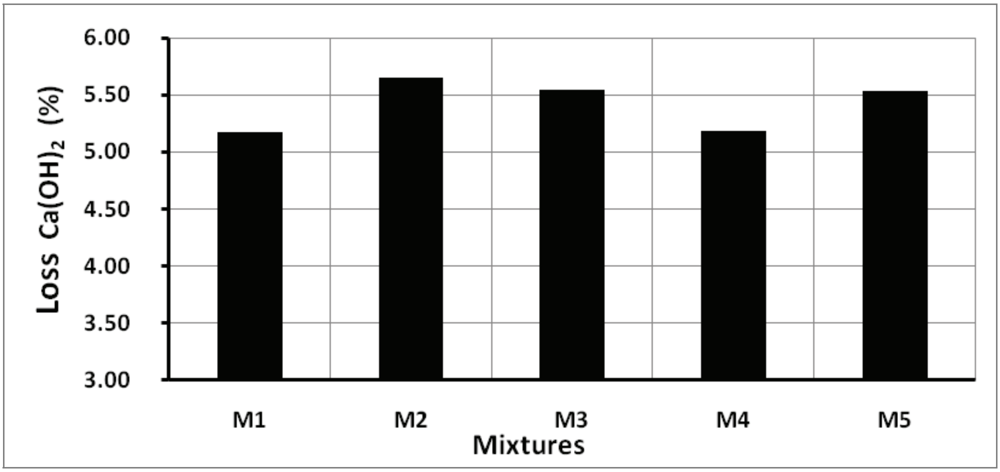

Fig. 10. $Ca(OH)_2$ mass loss during the thermo-gravimetric analysis of the studied mortars

The thermogravimetric analysis are carried out after 28 days on mortars in order to estimate the energies of activation of the dehydroxylation reaction, which makes it possible to quantify the Portlandite by determination of the weight loss of $Ca(OH)_2$ present in the mortars studied.

The results presented in the figure 10, clearly show that the binary mortar (M2) with 20% pozzolan and the quaternary mortar (M4) with pozzolan dominance contain respectively the lowest quantities of portlandite (5.174% and 5.17%), which highlights the pozzolanic activity which is characterized by two distinct aspects (Majumda et al. 1977; Dron 1978; Massazza and Costa 1979; Barret and Menetrier 1997). The total amount of calcium hydroxide that a pozzolan is able to bind and the calcium hydroxide binding kinetics.

In addition, the binary mortar with slag (M3) which records a loss of calcium hydroxide close to that of pozzolan, proves that the slag can also fix portlandite, some authors have shown that a mole of slag consumed 2.6 moles C-H (Biernacki et al, 2002). Nevertheless, mortars with slag dominance (M2 and M5) show a less active pozzolanic power relative to mortars with pozzolan dominance.

3.4. MERCURY MICROPOROSITY (MIP) FOR STUDIED MORTARS

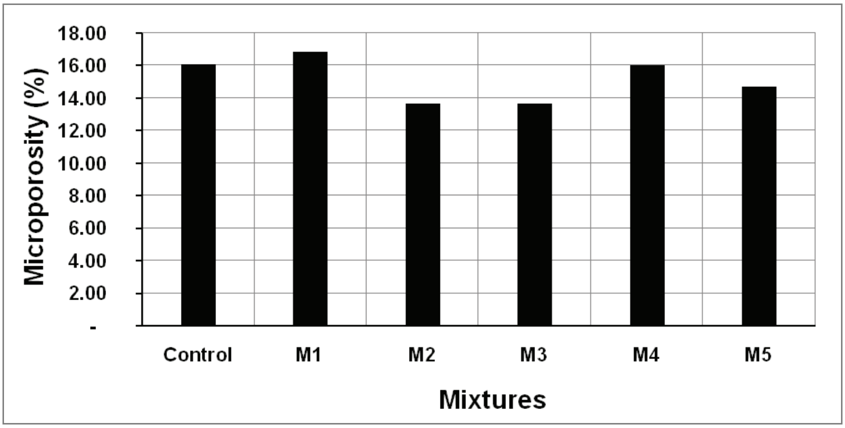

Fig. 11. Variation of the microporosity (MIP) of the mortars studied

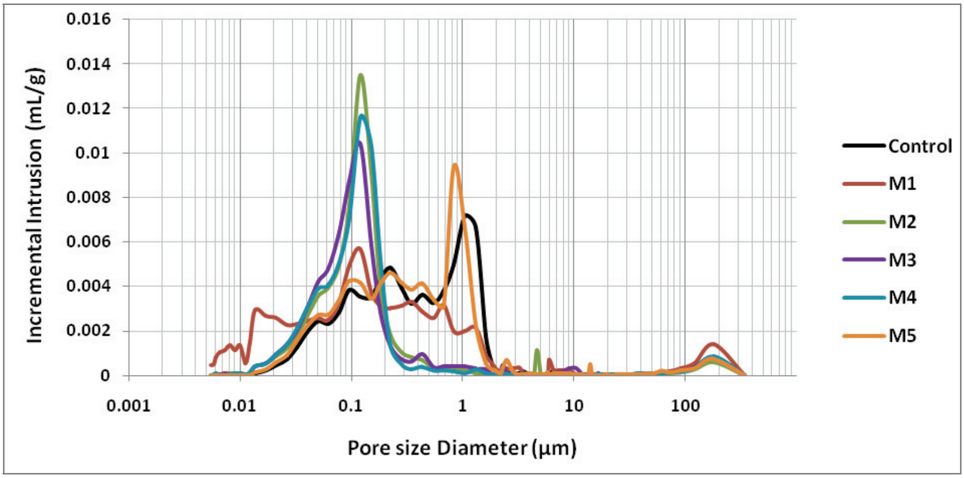

Fig. 12. Porous distribution of studied mortars

The analysis of the results obtained by the mercury microporosity test (Table 13) clearly shows that the binary mortar (M2) with 20% slag develops a less porous structure (13.65%) with pores of small sizes varying from 0.01 µm to 1 µm (Fig. 12) distributed over its entire volume. The binary mortar (M1) with 20% pozzolan develops a structure with a higher microporosity (16.85%) compared to that of a control mortar (Fig. 11).

The microporosity of the quaternary mortars (M4 and M5) is lower compared to the control mortar, which shows the interest of the interaction of slag and limestone additions with pozzolan, which tend to minimize the high porosity developed by pozzolan. It is found that the limestone plays a role of preferential nucleation site during the hydration reactions of the cement, generating a better distribution of hydrates, in particular of portlandite, in the hardened paste (Caré et al. 2000) and the interaction (slag-limestone) is very beneficial for the development of good compactness.

3.5. COMPRESSIVE STRENGTH OF THE MORTARS WITH MINERAL ADDITIONS

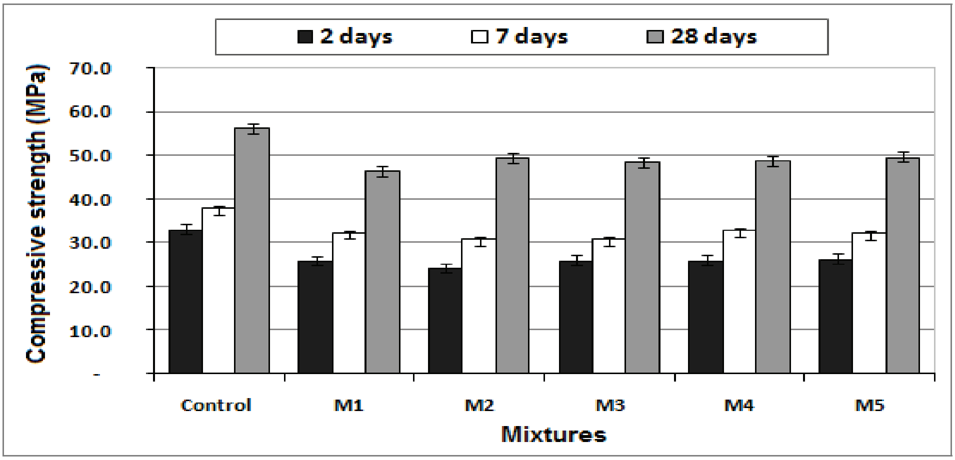

Fig. 13. Compressive strength of the mortars at the age of 2, 7, and 28 days (control, M1, M2, M3, M4 and M5)

The analysis of the results (Fig. 13) clearly shows that the compressive strength of the mortar control is better compared to the mortars with additions. Although, all the mortars with additions developed resistances at 28 days higher than the value that represents the guaranteed minimum resistance (40 MPa) for a CEMII/A. At the age of 2 days and 7 days; the binary mortar (M1) with 20% pozzolan offers better compressive strength compared to the mortar (M2) with 20% slag. Quaternary mortars with limestone filler show that it increases the mechanical resistance at young age which has been reported by some authors (Amouri, 2009) due to the accelerating effect and the effect of load limestone.

On the other hand, at the age of 28 days, the resistance of the binary mortar (M2) with 20% of slag evolution is better compared to the binary mortar (M1) with 20% of pozzolan, which highlights a significant reactivity of long-term slag as well as the maturity and stability of the microstructure with better compactness according to the results of the microporosity. The quaternary mortar (M5) shows better resistance compared to the binary mortar which explains the positive effect of the interaction between additions.

Regarding the variation of the individual results around the averages, we note that the standard error is minimal and does not affect the results

3.6. RESISTANCE ACTIVITY INDEX (RAI)

A mechanical approach allows us to estimate the activity of the additions through the results of the mechanical resistance at 28 days.

Résistance activity index (RAI) defined as the ratio: A/B × 100.

A: The average compressive strength of cubes for pozzolan mixes (MPa).

B: The average compressive strength of cubes for mixes du clinker alone (MPa).

The (ASTM C618, 93) standards require a minimum activity index of 75% at 28 days.

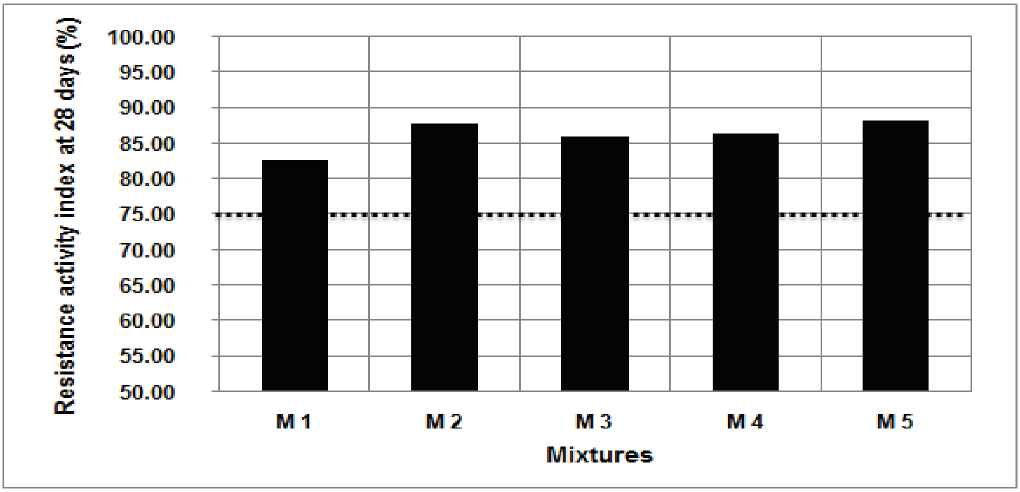

Fig. 14. The variation of the resistance activity index (RAI) of mortars studied at 28 days

The estimate of the resistance activity index presented in (Fig. 14) shows that the best index is recorded for the binary mortar (M2) with 20% slag and the quaternary mortar (M5) with slag dominance respectively (87.90 and 88.13) which correlates with the results of the microporosity (MIP) which shows that the slag with a dominant rate is in binary or quaternary comopsition with limestone. This develops less porous structures with a good compactness which leads to develop better mechanical resistance.

4. CONCLUSIONS

The results obtained of the characterization of reactivity of mineral additions (pozzolan, limestone and slag) through the formulation of binary, ternary and quaternary pastes and mortars shows that the interest of 20% rate substitution of cement by mineral additions studied as natural and industrial mineral additions. These additions could produce economic, ecological and efficient mortars.

The physico-chemical and micro-structural characterization of reactivity of the mineral additions confirms that any addition alone does not always have the best performances in regards to all the properties at the same time (reactivity, porosity and strength).

The binary cement with 20% pozzolan (active additions) shows a better pozzolanic activity for the pastes compared to the slag, but without affecting the mechanical resistance which remains important and comparable to that of the control mortars at any age.

The binary cement with 20% slag (active additions) shows a better compactness and very low porosity compared to the pozzolan addition and the control mortar. The compressive strength is competitive with that of the control mortars, even comparable at the age of 28 days.

Characterization of the reactivity of the additions can be demonstrated by microstructural or mechanical approaches using experimental methods for the qualitative and quantitative analysis of hydration products and their distributions at the microstructure level. The X-ray diffractions shows the hydraulic and pozzolanic power of active additions (slag and pozzolan) through the quantification of hydrated calcium silicates and portlandite, which demonstrate almost identical mineral phases in nature and intensity.

Thermogravimetry is able to highlight the important pozzolanic activity of the Pozzolan compared to the slag. Also, mercury microporosometry clearly shows that the slag developes the best microstructures, less porous and more compact cement matrix. SEM images gives largely information on the nature of the hydrates morphology and their distribution. The chemical and microstructural variations influence directly the mechanical responses. Furthermore, It shows that microstructural investigations correlate with mechanical responses results, and the activity index gives an advantage of slag activity relative to pozzolan and demonstrates the positive effect of limestone inert fillers.

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