



# Journal of Materials and Engineering Structures

## Research Paper

### Study of Mortars Made with Natural and Artificial Pozzolans

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#### ARTICLE INFO

##### Article history :

Received : 19 October 2018

Revised : 7 December 2018

Accepted : 10 December 2018

##### Keywords:

Valorization

thermal activation

pozzolan

siltation

#### ABSTRACT

The objective of this work is to evaluate the thermal activation potential of clay soils, particularly the clay fraction of dam mud, for the production of artificial pozzolan which can be substituted to cement in concrete and mortars applications and also to make a comparative study with the natural pozzolan available in the area of Beni Saf in considerable quantities. The research study is carried out on three mortars 10, 20 and 30% of natural pozzolan of Beni Saf and artificial pozzolan from the mud calcined at 850 ° C / 3 hours of the dredged sediments of the dams of Fergoug and Ouizert, and on a control mortar without additions for the need of comparison. Several physical, mechanical, microstructural and sustainable tests have been carried out to carry out this research study: maneuverability in the fresh state, impeded shrinkage, mechanical performance, absorption, acid attack (5% CH<sub>3</sub>COOH, 5% HNO<sub>3</sub> and 5% H<sub>2</sub>SO<sub>4</sub>), mass loss and pH reading follow-up in the hardened state. Compressive strengths indicate that pozzolan-based mortars have the best results in particular over the long term, whereas the results of acid attack tests show that the calcined mud has a significantly greater influence than natural pozzolan.

## 1 Introduction

Algeria possesses an appreciable quantity of natural pozzolanic material of volcanic origin, which extends sporadically over a distance of 160 km, between the Algerian-Moroccan border and the Sahel of the city of Oran. The country also has numerous dams in a state of advanced siltation, thus containing considerable quantities of silt capable of giving artificial pozzolan after heat treatment. Natural and artificial pozzolans are used only partially in the manufacture of cements for mortars. Some researchers have reported some improvements in the mechanical and thermal characteristics of pozzolanic mortars. The valorization of pozzolans can certainly contribute to the reduction of their impact on the environment. This impact may be that due to the dredged sediments or that resulting from the production of cement that is accompanied by very intense greenhouse gas emissions, [1], [2], [3], [4].

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Studies on the incorporation of natural and artificial pozzolans as partial replacement of cement have shown the possibility of using these materials, available in large quantities in nature for natural pozzolan and in dams for artificial pozzolan, for improving the performance of mortars and concretes with minimal environmental impact, [5], [6], [7], [8] et [9].

The use of Portland cement with pozzolan generates a reaction between the calcium hydroxide, produced by the hydration of Portland cement and the amorphous  $\text{SiO}_2$  of the glassy phase of pozzolan. This leads to the formation of a larger amount of calcium silicate hydrate [C-S-H], of second generation, and thus to an increase in strength and density of the mortars obtained, [10], [11], [12].

The selected vase submitted to the research study comes from two dams of the wilaya of Mascara: Fergoug and Ouizert. These two dams have reached a critical level of siltation, for example, the Fergoug dam, which is overflowed at more than 80% of its capacity, is cleared per year. 6 million cubic meters of sediment and must be extracted by National Agency of Dams and Transfers. So the natural mud is also available in considerable quantities near two cement plants (Oggaz and Zahana). The present work aims at examining the use of natural and artificial pozzolans as partial substituent for Portland cement in mortar and consequently to evaluate their effects on the mechanical performance and durability of the mortars obtained. For this purpose, some physical, chemical, mechanical and durability tests (acid attack with measurements of mass loss, pH monitoring of the aggressive solutions, water absorption and restrained shrinkage) were performed. The tests carried out on pozzolanic mortars showed that the mechanical performance of pozzolanic mortars and their durability are improved as compared with those of control mortar, which was formulated without pozzolan.

## 2 Materials and methods

### 2.1 Cement

The cement used in all the tests is the white Malaki Portland limestone cement CEM II / A-L 52.5N, from the Lafarge cement plant in Oggaz, near the town of Mascara (Algeria), and manufactured according to the Algerian standard, [13]. The physical properties and the mineralogical composition of the cement used are shown in tables 1 and 2. Its chemical composition is given in table 3. We don't choose a CEM I that contains clinker exclusively because currently, throughout the national territory, for ecological and especially economic reasons, Algerian cement plants no longer produce CEM I.

**Table 1 - The Physical Properties of Cement**

Blaine specific surface ( $\text{cm}^2/\text{g}$ )	Apparent volumetric mass ( $\text{g}/\text{cm}^3$ )	density	Start of Setting Time (min)	End of Setting Time (min)	Expansion "Le Chatelier" (mm)
4610	1.26	3.12	178	233	0.90

**Table 2 -The Mineralogical Composition of Cement (%)**

$\text{C}_2\text{S}$	$\text{C}_3\text{S}$	$\text{C}_3\text{A}$	$\text{C}_4\text{AF}$
22.50	63.00	5.00	9.25

**Table 3 - The Chemical Composition of Cement (%)**

CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	MgO	LOI
65,89	21,4	2,96	0,52	1,72	0,41	0,03	0,59	7,20

### 2.2 2.1.2 Natural pozzolan

First, free water was removed by a drying process at the temperature of 105 °C, then the pozzolan was finely ground, as shown in figure 1. The physical properties and the chemical composition of natural pozzolan, from the town of Beni-Saf, are shown in tables 4 and 5, respectively.



Fig. 1 – (a) Natural pozzolan slag before grinding; (b) natural pozzolan powder after grinding.

Table 4 - The Physical Properties of Natural Pozzolan.

Blaine specific surface (cm <sup>2</sup> /g)	Apparent volumetric mass (g/cm <sup>3</sup> )	density	Absorption (%)	Porosity (%)	Humidity (%)
4255	0.98	2.65	58,70	57,10	2,50

Table 5 - The Chemical Composition of Natural Pozzolan (%).

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	PF
46.83	17.45	8.36	10.20	3.88	0.36	1.40	4.32	4.79

The scanning electron microscopy indicated that natural pozzolan has a complex microstructure. The X-ray spectral analysis showed the presence of larger or smaller quantities of elements such as: Si, Al, Na, K, Mg, Fe and Ca, respectively in descending order, figure 2. The X-ray diffraction showed the mineralogical composition of pozzolan which contains crystalline minerals and a vitreous phase, figure 3. What interests us from a DRX diagram is the crystalline phase, since it allows the formation of C-S-H. The minerals of the crystalline phase are shown in the legend of figure 3.

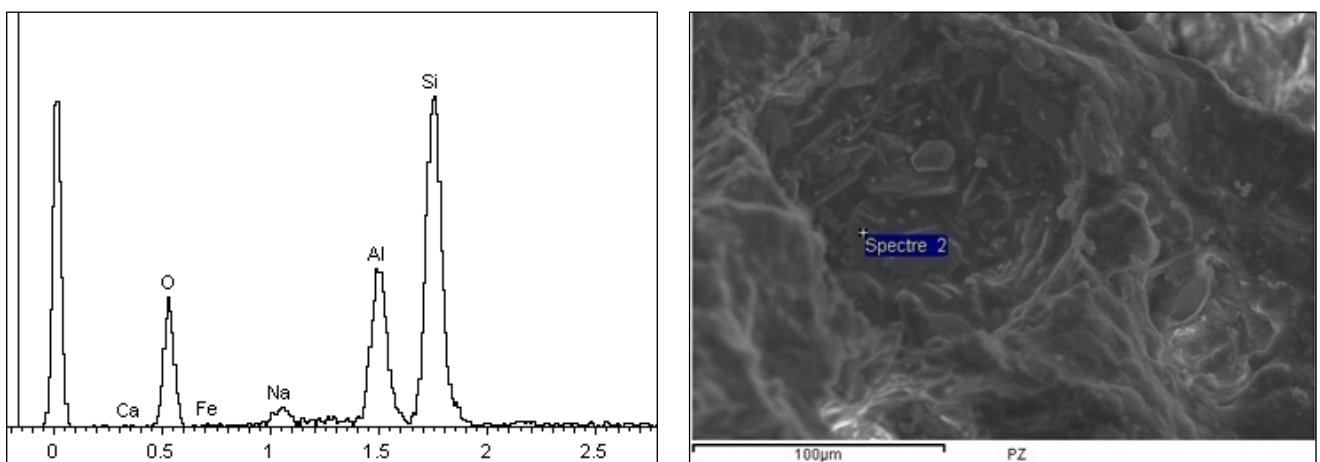


Fig. 2 - Micrography and X-ray spectra of natural pozzolan.

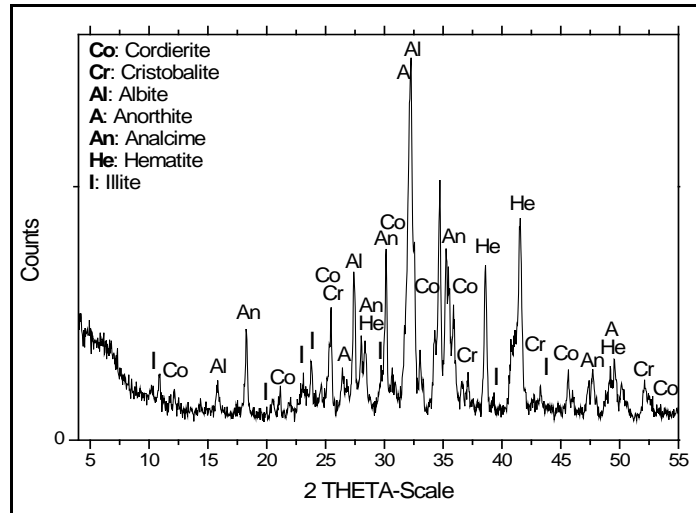


Fig. 3 - X-ray diffraction of natural pozzolan.

2.3 Sand

In the experimental study of the present work, crushed sand of fraction 0/3 was used from the quarry of Tizi, in the Wilaya of Mascara (Algeria). The physical properties of that sand are given in table 6; its granulometric analysis is given in Fig. 4.

Table 6 - The physical properties of sand

Apparent volumetric mass (g/cm <sup>3</sup> )	Absolute volumetric mass (g/cm <sup>3</sup> )	Sand equivalent (%)	Modulus of fineness
1,380	2,600	75	2.27

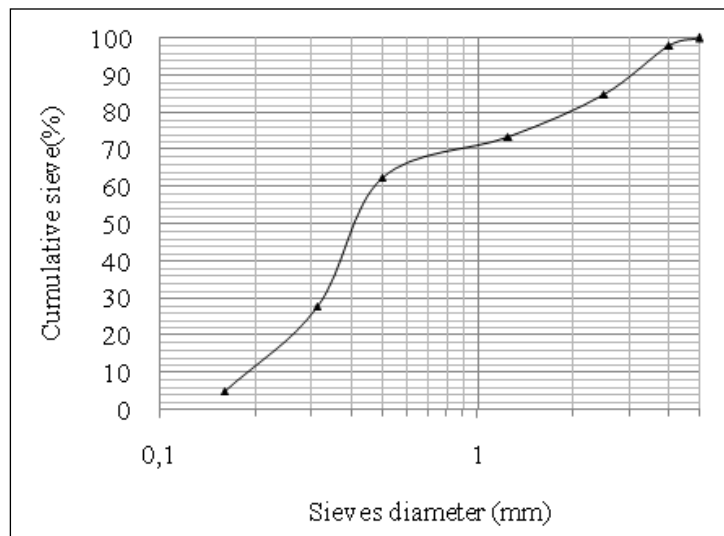


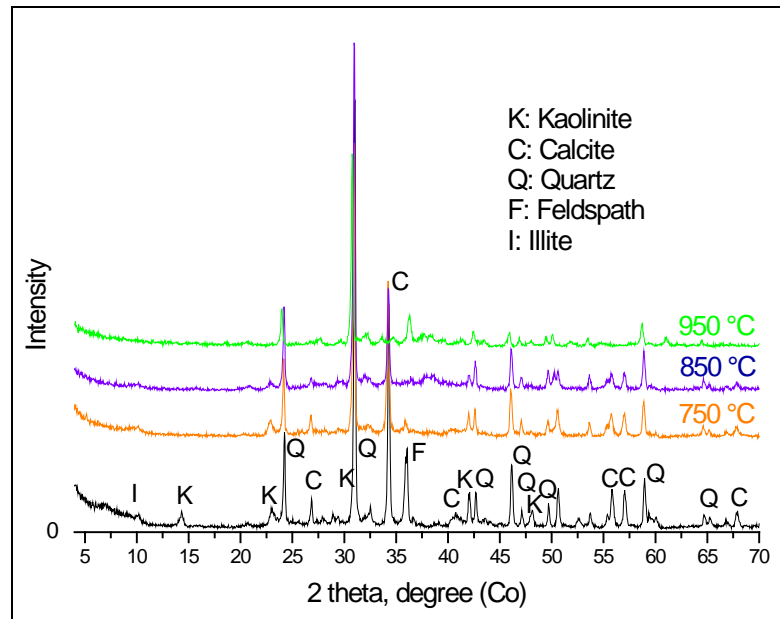
Fig. 4 - Granulometric analysis curve of sand.

2.4 The calcined mud

All sludge used in our study was calcined at a temperature of 850°C for a period of 3 hours since according to the bibliographic study, the optimal temperature calcination of the mud is around 850°C during a duration of 3 hours. The physical characteristics of calcined muds of the Fergoug and Ouizert dams are shown in table 7. The X-ray diffraction showed the mineralogical composition of pozzolan which contains silica, kaolinite, calcite, illite (traces), figure 5.

**Table 7 - The Physical Characteristics of Calcined Muds**

	Fergoug mud	Ouizert mud
Apparent volumetric mass (g/cm <sup>3</sup> )	0.89	0.82
Absolute volumetric mass (g/cm <sup>3</sup> )	2.65	2.55
Blaine specific surface (cm <sup>2</sup> /g)	4650	4630



**Fig. 5 - X-ray diffractogram of natural and calcined mud.**

**2.4.1 Chemical compositions of different kinds of mud used**

The chemical and mineralogical compositions of the vases of Fergoug and Ouizert dams are illustrated in the following tables 8 and 9. The X-ray fluorescence showed three oxides SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> which are essential elements in the manufacture of calcium silicate hydrate (C-S-H) gels upon hydration of cement. The base composition of the cements is a mixture of silicates (C<sub>3</sub>S, C<sub>2</sub>S) and calcium aluminates (C<sub>3</sub>A, C<sub>4</sub>AF), resulting from the combination of lime (CaO) with silica (SiO<sub>2</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>), and iron (Fe<sub>2</sub>O<sub>3</sub>). The required lime is provided by limestone rocks, alumina, silica and iron oxide by clays. The main cement compounds C<sub>3</sub>S, C<sub>2</sub>S, C<sub>3</sub>A, and C<sub>4</sub>AF react to form new insoluble compounds that cause the setting and progressive hardening of the material. There is also a small increase in the amount of lime (CaO) as a function of calcination temperature of the sludge because Around 800 to 850 ° C, we have the phenomenon of decarbonation of CaCO<sub>3</sub>, and consequently the increase of the CaO rate. Indeed, the thermogravimetric study is very useful for evaluating the changes in the composition of the thermal treatment mud, but unfortunately, we do not have an ATG / ATD device. Dam sediments are considered artificial pozzolan because they are composed mainly of silica (SiO<sub>2</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>) and ferrite (Fe<sub>2</sub>O<sub>3</sub>), the same constituents as natural pozzolan. In addition they are calcined. Depending on the oxides composition of the dam sediments, the silica content (SiO<sub>2</sub>> 41%) is higher than that of the alumina (Al<sub>2</sub>O<sub>3</sub><16%). The percentage of CaO in Ouizert waste is much lower than the percentage of calcite because it is very difficult to keep the CaO without it being carbonated, since the CO<sub>2</sub> level is very high in the atmosphere. CaO reacts with CO<sub>2</sub> to form CaCO<sub>3</sub> (Calcite).

The sum of the mineral waste of Ouizert and Fergoug are 91.4 and 92.7 percent, it is not 100 percent because the device used for the mineralogical analysis gave us 11 species, but there are other minerals with very low rates that the device cannot detect. In Table 9, it is X-ray Diffraction of Calcined Mud. The XRD analyzes were carried out at the Lafarge-Oggaz cement plant. The DRX device has a program that gives the percentage of minerals.

**Table 8 - The Chemical Composition of Calcined Muds (%)**

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	LOI
Natural Mud	41.40	7.82	5.02	17.47	3.12	0.32	1.36	1.17	22.84
Fergoug Calcined Mud	51.69	15.49	7.53	18.06	3.08	0.23	2.99	0,41	1.87
Ouizert Calcined Mud	49.55	15.21	6.37	18.32	2.23	0.21	2.76	0.52	4.48

**Table 9 - X-ray Diffraction of Calcined Mud**

	Calcite	Dolomite	Quartz	Illite	Kaolinite	Chlorite	Pyrophyllite	Albite	Microcline	Diaspore	Topaz
Ouizert mud (%)	13.56	1.81	21.64	32.7	10.47	0.00	0.00	4.27	5.68	0.41	0.86
Fergoug mud (%)	26.10	6.48	18.61	26.5	8.72	0.10	0.10	2.70	1.35	0.88	1.16

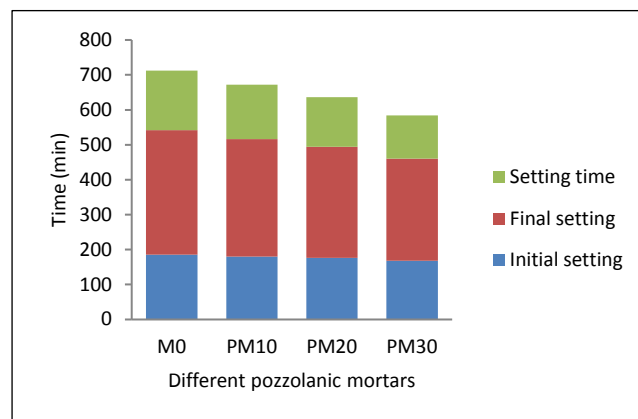
**2.5 Preparation of mortars**

Mortars intended to be used in the preparation of test specimens of dimensions (4 x 4 x 16) cm<sup>3</sup> were formulated according to standard NF EN 196-1, [14]. The control mortar M0 (without pozzolana) is an ordinary mortar. The pozzolanic mortars were made from normal mortar, with partial replacement of cement by pozzolana, with the ratio W/C = 0.5. The substitution percentages of cement by the pozzolan were 10%, 20%, and 30%, for all pozzolanic materials. We don't compare our results with an inert addition because they do not have the same role, the inert additions play the role of filling micropores against our additions have a pozzolanic action with portlandite. A superplasticizer (2.6% by mass of the cement), type Sika viscocrete tempo 12, was used for the formulation of all the mortars. It is a waterproof, versatile, new generation, non-chlorinated water-based super-plasticizer, based on acrylic copolymer. The specimens were removed from their molds after 24 hours and stored in lime-saturated water, at a temperature of 20 ± 2 °C. The different mortars prepared are:

M0 : mortar control (0% addition); PM10, PM20 et PM30 mortars of 10, 20 et 30% of natural pozzolan respectively; FM10, FM20 et FM30 mortars à 10, 20 et 30% of Fergoug dam mud respectively and OM10, OM20 et OM30 mortars à 10, 20 et 30% of Ouizert dam mud respectively.

**2.5.1 Normal consistency and setting time of mortars**

The results of the characterization physical testing of standardized cement pastes with and without natural pozzolana are given in figure 6.



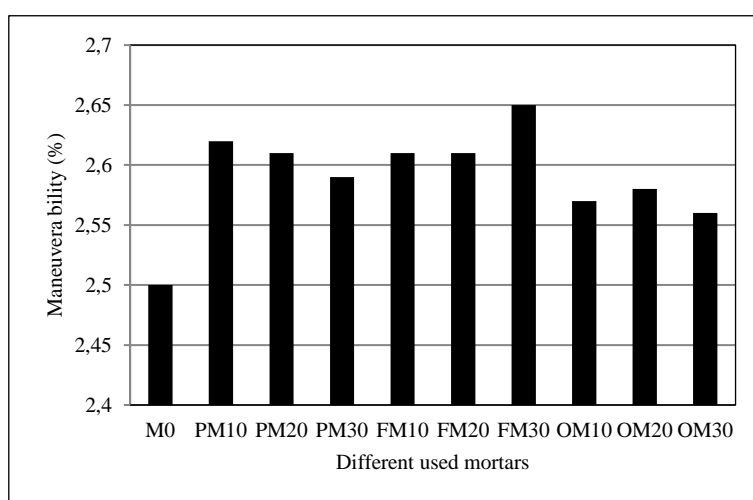
**Fig. 6 - Setting of mortars.**

The test for the *initial* and *final setting times of mortar*, according to standard NF EN 196-3, [14], was carried out for all the other mortars, namely PM10, PM20 and PM30, in order to have a consistency close to that of standard mortar without pozzolan M0. The increase in the normal consistency of pozzolanic mortars has a negative impact on their mechanical properties, in this case the compressive strength. In figure 6, it is clearly noted that with the same consistency, the *initial* and

*final setting times* decrease with increasing natural pozzolan dosages. This is quite normal, because pozzolanic mortars with greater fineness need a larger normal consistency. The increase in the normal consistency of pozzolanic mortars may be explained by the progressive augmentation in the specific surface area of the binder, which will naturally lead to a greater need for water in order to wet the whole paste. The increase of the consistency can be explained by the effect of the specific surface as well as the absorption of mixing water by the pozzolan because of its porous structure.

### 2.5.2 Workability of fresh mortars

Workability of fresh mortars was determined by means of a shaking table, according to standard ASTM C 1437, [15]; the same amount of water was used for the preparation of all paste mixes (Water / Binder = 0.5). Figure 7 illustrates the influence of the additions on the workability of the mortars. According to this figure, the incorporation of mineral additions affects the workability of fresh mortars. Indeed, the workability of all pozzolanic mortars increases, and this will make the mortar mixture more plastic or creamier. The workability of pozzolanic mortars decreases as the substitution level of cement by natural and artificial pozzolans goes up. This workability decline in pozzolanic mortars is reflected by the proportional increase in the specific surface area of binders, and there is therefore an additional need for water. Portland cement mortar with no additions has less workability than pozzolan mortars.



**Fig. 7 - Influence of the maneuverability of used mortars.**

### 2.6 Mechanic performances

Samples of dimensions (4x4x16) cm<sup>3</sup> with the different sludge used were manufactured. The samples were stored in water for the duration of the maturity at room temperature of the laboratory. Mechanical performance tests (compression and bending) were performed according to ASTM C 109, [16].

### 2.7 Action of acid solutions

Mortar specimens of dimensions (50x50x50) mm<sup>3</sup> were used for assessing the resistance of mortar to three different acids, namely CH<sub>3</sub>COOH, H<sub>2</sub>SO<sub>4</sub> and NHO<sub>3</sub>. These samples were made with Portland white cement CEM II / A-L 52.5N Malaki from the Lafarge cement plant in Oggaz, in the town of Mascara (northwestern Algeria); then, they were mechanically compacted using a shock table. The preparation and preservation procedures were done according to the standard NF EN 196-1, [14]. After 28 days of curing in a lime-saturated water solution, at the temperature 20 ± 2 °C, the specimens were weighed (M1) and then immersed separately in three different chemical solutions, namely 5% acetic acid (CH<sub>3</sub>COOH), 5% sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and 5% nitric acid (NHO<sub>3</sub>). The resistance of the samples to the chemical aggressions of these solutions was evaluated according to the standard ASTM C 267-01, [17]. First, the test pieces were washed and cleaned three times with fresh water to remove the altered mortar and then they were allowed to dry for half an hour in order to weigh them with a 0.01 g precision balance to determine their masses (M2) after the chemical attack by the three acids. The weight loss of

the test specimens was calculated after 7, 14, 21 and 28 days of immersion in the chemical solutions, which were renewed every seven days.

This calculation was performed using the following equation:

$$\text{loss of mass (\%)} = \frac{M1 - M2}{M2} \times 100 \quad (1)$$

M1: mass of the sample before immersion in the aggressive solution, M2: mass of the sample after immersion in the aggressive solution.

### 3 Results and analyses

#### 3.1 Density of mortars

Figure 8 shows the evolution of the density of mortars as a function of the duration of immersion in water, after 3, 7, 21 and 28 days. An increase in the density was noted for all mortars, and this is certainly due to the important water demand of the mortars during the first hydration phase. It was also noted that the density of all mortars was relatively higher than that of the control mortar, which is probably due to the effect of the additions; this what makes the matrix denser. It was found that the mortar OM30 (Ouizert mortar with 30% of mud) has the largest bulk density, at all ages.

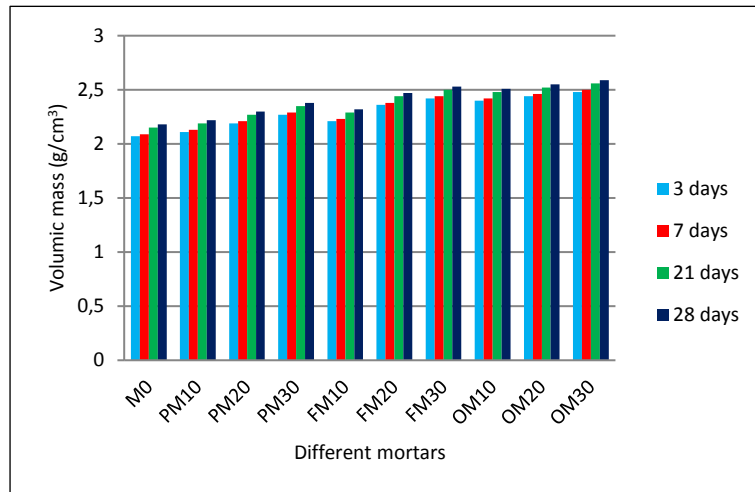


Fig. 8 - Volumic masses evolution of used mortars.

#### 3.2 Mechanical performance

##### 3.2.1 Tensile strengths of mortars

The immersion period of these test pieces in water, at the ambient temperature of  $20 \pm 2$  °C, was 45 days. The results of the flexural strength testing are summarized in figure 9. This figure 9 (a) shows that the flexural strengths of the mortars PM10 and PM20 are close to that of the control mortar M0. For maturities exceeding 45 days, the tensile strength values would be greater than that of the control mortar M0, because the pozzolanic reaction is slow and requires more time to be fully completed. Figure 9 (b) indicates that the flexural strength of the mortar FM10 is 10% higher than that of the control mortar. It is worth noting that the mud from the dam of Fergoug, calcined at 850 °C, has an important pozzolanic reactivity since its flexural strength exceeds that of the control mortar.

In addition, figure 9 (c) also shows that the flexural strength of the mortar OM10 is higher than that of the control mortar M0. It thus seems that the mud from Ouizert dam can be used as an addition in mortars. Figure 9 (d) shows a clear comparison between the effects of different pozzolans on the flexural strength of mortars. The incorporation of calcined mud, with 10% substitution (FM10 and OM10), gives flexural strengths higher than those of all other mortars, including the control mortar. This fact allows us to say that when the particles of calcined mud are well deflocculated by the superplasticizer, they promote the hydration reaction. It is also observed that the flexural strength of the 10% natural pozzolan mortar (PM10) is substantially similar to that of the control mortar M0. Therefore, all pozzolanic mortars have good flexural strengths.



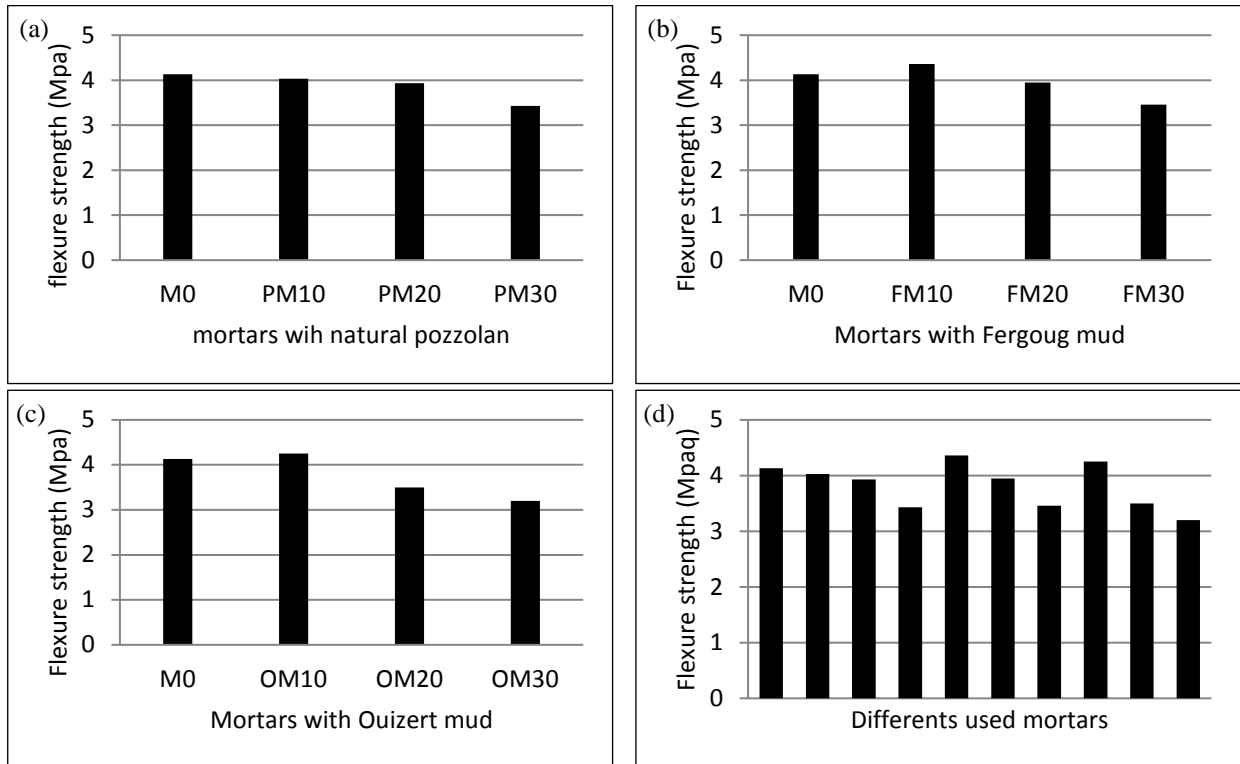


Fig. 9 - Used additions effect on the flexure strength.

### 3.2.2 Compressive strengths of mortars

Evolutions of the compressive strengths as a function of the calcined mud and pozzolan contents are illustrated in figure 10.

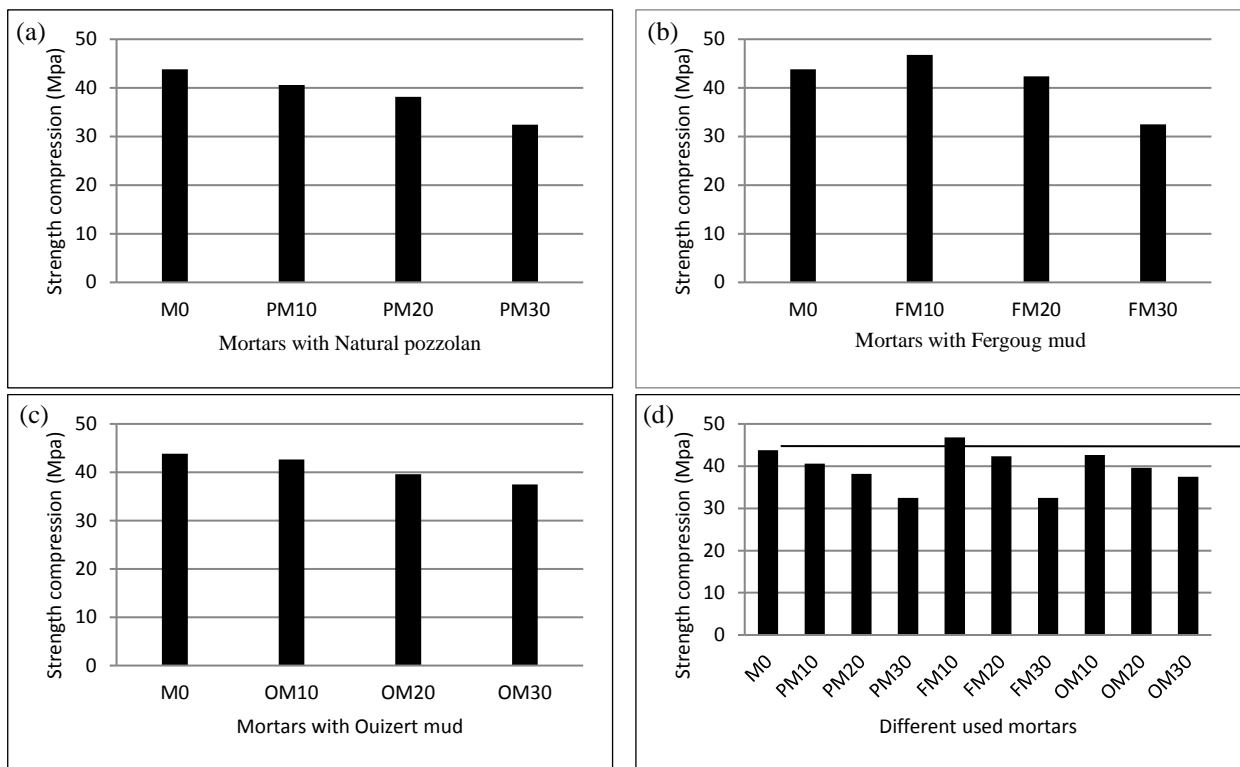


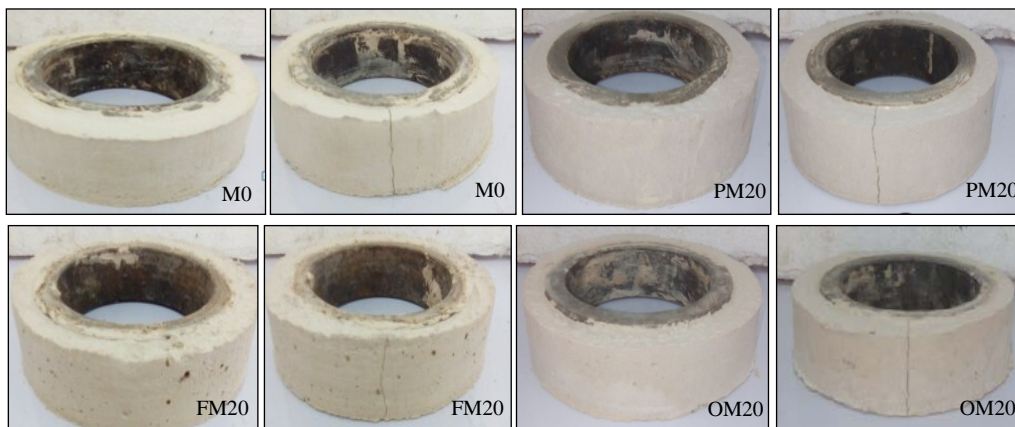
Fig. 10 - Used additions effect on the compression strength.

The immersion period of these test pieces in water was 45 days, with an ambient temperature of  $20 \pm 2$  °C. Figure 10 (a) indicates that the compressive strength of the mortar PM10 is 20% lower than that of the control mortar. It then decreases by 30% and 60% for the mortars PM20 and PM30, respectively, still with respect to the compressive strength of the control mortar. This strength decline is mainly attributed to the slow activity of natural pozzolana. This phenomenon may be explained by the interaction between the reactive silica, which is encountered in the vitreous part of natural and artificial pozzolans, and the  $\text{Ca}(\text{OH})_2$  released during the hydration of cement. The pozzolanic reaction is not predominant at young age, and this leads to less intense hydration at young age. As a result, weak compressive strength are generated. Figure 10 (b) shows that the compressive strength of the mortar FM10 is 7.31% higher than that of the control mortar, and those of mortars FM20 and FM30 are lower by 12.94 and 25.91%, respectively, relative to the control mortar. It is worth noting that, at 45 days, the strength of the 10% calcined mud mortar from the dam of Fergoug (FM10) has already exceeded that of the control mortar. Figure 10 (c) shows that the compressive strength values of mortars OM10, OM20 and OM30 decrease by 2.60%, 9.59% and 14.38%, respectively, with respect to that of the control mortar. At the age of 45 days, the strength of the 10% calcined mud mortar from Ouizert dam approaches that of the control mortar. Figure 10 (d) suggests that the compressive strengths of mortars decrease considerably with the increase in the pozzolan substitution levels. It is easy to notice that among the pozzolanic mortars, PM10, FM10 and OM10 (all containing 10% of pozzolan) have the best compressive strengths. The mud from Fergoug dam, with 10% substitution level (FM10), seems to give the highest value of the compressive strength; this value exceeds even that of the control mortar (M0) by 5.74%. These results were obtained at the age of 45 days only.

At longer maturities, one should expect that these strengths be improved as compared to that of the control mortar, because the pozzolanic reactions are slow. These findings indicate the improvement of the compressive strengths of mortars, in which cement is replaced by pozzolanic materials; these results have already been confirmed by several researchers, such as [5], [8], [11], [9].

### 3.3 Restrained shrinkage test

This test consists of visually assessing the shrinkage and swelling of various mortar specimens, of dimensions  $(4 \times 4 \times 16)$   $\text{cm}^3$ , over a period of 45 days. Figure 11 shows the different test specimens M0, PM20, FM20 and OM20 before and after cracking. After 45 days of storage in the open air, cracks appeared in all the mortars. It is clearly noted that pozzolanic mortars present smaller cracks than the normal mortar, without pozzolana. Therefore, the pozzolanic materials are beneficial since they make it possible to reduce the cracking due to the restrained shrinkage.



*Fig. 11 - Samples subjected to shrinkage test before and after cracking*

### 3.4 Durability

#### 3.4.1 Acid attacks

Figures 12, 13 and 14 display the evolution of mortar mass loss as a function of the immersion period in the aggressive solutions of 5%  $\text{CH}_3\text{COOH}$ , 5%  $\text{HNO}_3$  and 5%  $\text{H}_2\text{SO}_4$ .

3.4.1.1 Acetic acid

The variations in the mass loss of the mortars immersed in the 5% acetic acid solution ( $\text{CH}_3\text{COOH}$ ) are illustrated in figures 12. It is clear that at 28 days, the weight losses of natural pozzolan mortars are lower than those of control mortar, figure 12 (a). Mortars MP10 and MP20 have the lowest weight loss. Thus, it can be said that natural pozzolan is beneficial because it offers better resistance to the aggression of acetic acid.

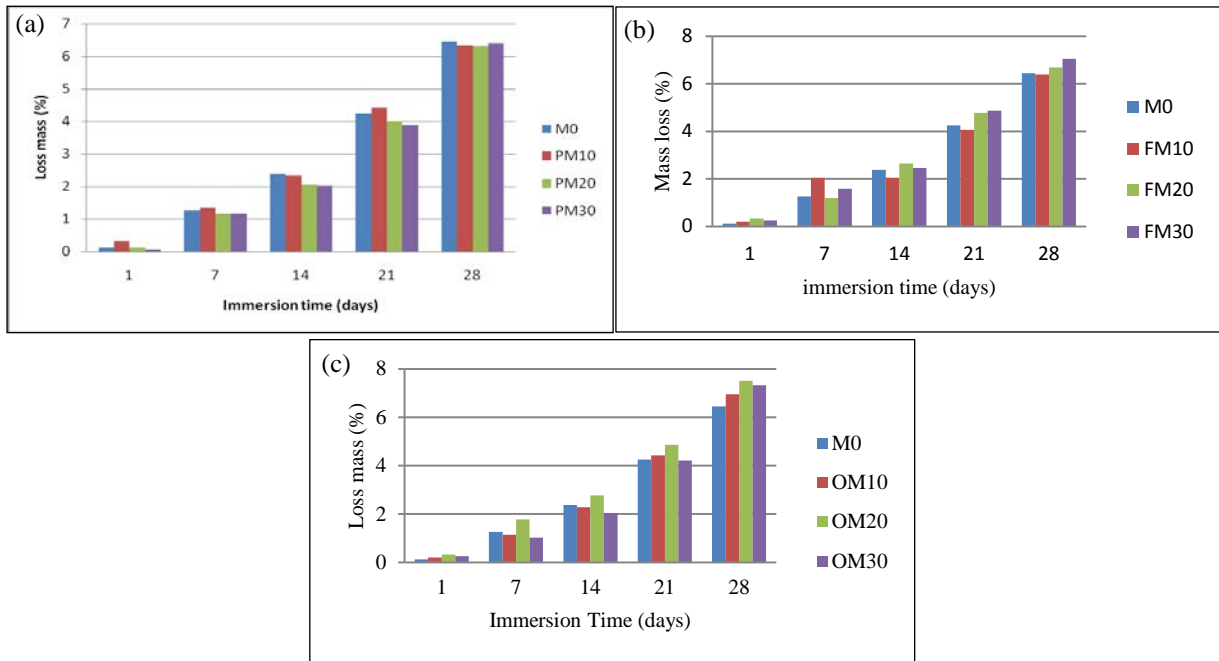


Fig. 12 - Loss mass of different mortars in 5%  $\text{CH}_3\text{COOH}$ .

3.4.1.2 Nitric acid

The variations of the mass loss with time of the immersed mortars in the 5% nitric acid solution ( $\text{HNO}_3$ ) are illustrated in figure 13.

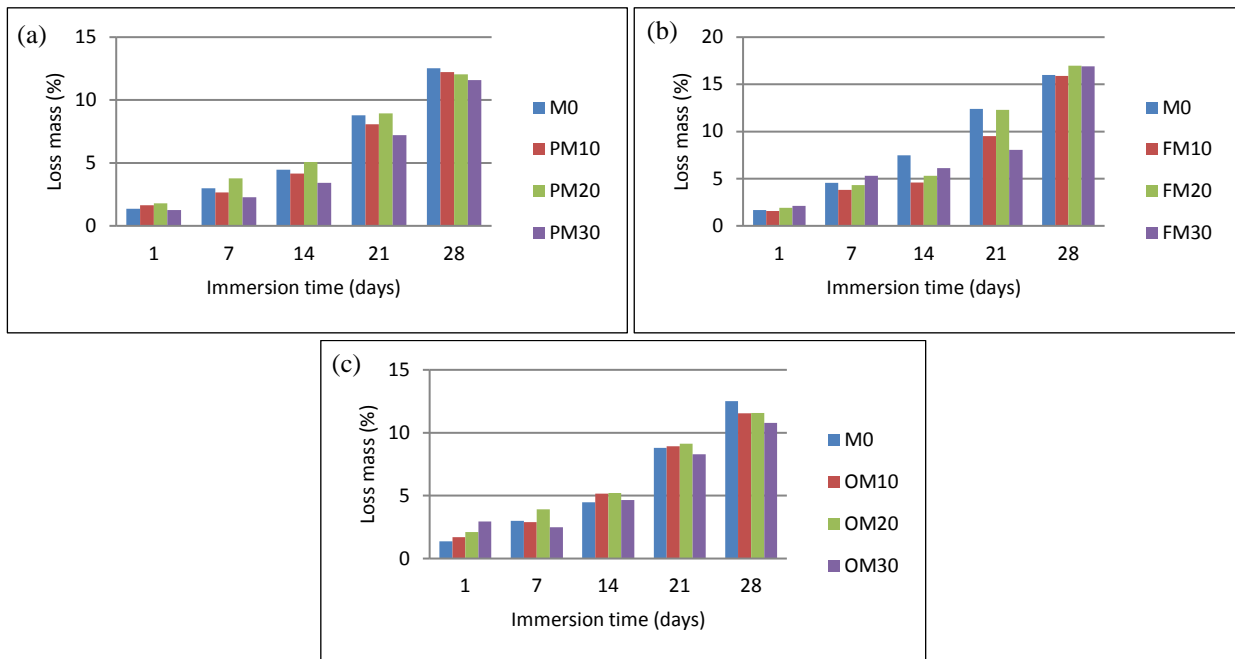


Fig. 13 - Loss mass of different mortars in 5%  $\text{HNO}_3$

It is clear that natural pozzolana and calcined mud from Ouizert dam have less weight losses as compared to the control mortar, after 28 days of immersion in that solution, figures 13 (a) and (c). One can easily observe that the mortars with calcined mud from Ouizert dam have the lowest weight loss. The natural pozzolan and calcined mud from Ouizert dam are therefore beneficial because they give greater resistance to the aggressive nitric acid.

3.4.1.3 Sulfuric acid

The variations of the mass loss of mortars immersed into the 5% sulfuric acid solution are illustrated in figure 16.

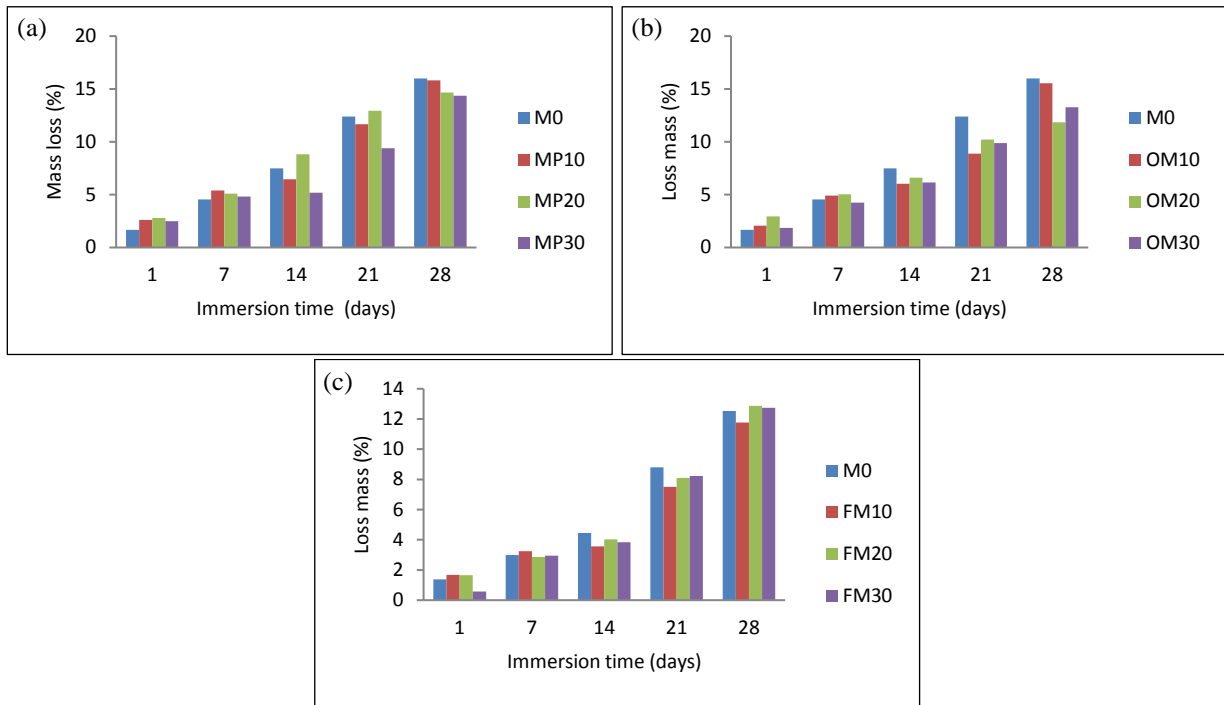


Fig. 14- Loss mass of different mortars in 5% H<sub>2</sub>SO<sub>4</sub>

For the 5% H<sub>2</sub>SO<sub>4</sub> aggressive solution, it is easy to notice that natural pozzolana and the calcined mud from Ouizert dam show smaller mass losses as compared to that of the control mortar, after 28 days of immersion, figures 14 (a) and 14(c). One can easily note that the mortars with calcined mud from Ouizert dam exhibit the lowest mass losses. Therefore, natural pozzolana and the calcined mud from Ouizert dam are beneficial since they are more resistant to the aggressive sulfuric acid solution.

3.4.1.4 Comparison of different mortars exposed to acid attacks

The comparison of the mass losses of the different mortars immersed into the aggressive solutions used in this study is illustrated in figure 15.

It can be seen that, for all replacement levels (PM10, PM20 and PM30), natural pozzolan resists fairly well to all acid attacks, figure 15 (a). The calcined mud from Ouizert dam resists well to nitric and sulfuric acids. The mud from the dam of Fergoug resists poorly to acid attacks.

It is also noted in figure 15 that sulfuric acid is the most aggressive, followed by nitric acid, and acetic acid comes last. Natural pozzolan seems to be the most beneficial pozzolanic material that resists to the aggressive acids, the authors [19] found the same result.

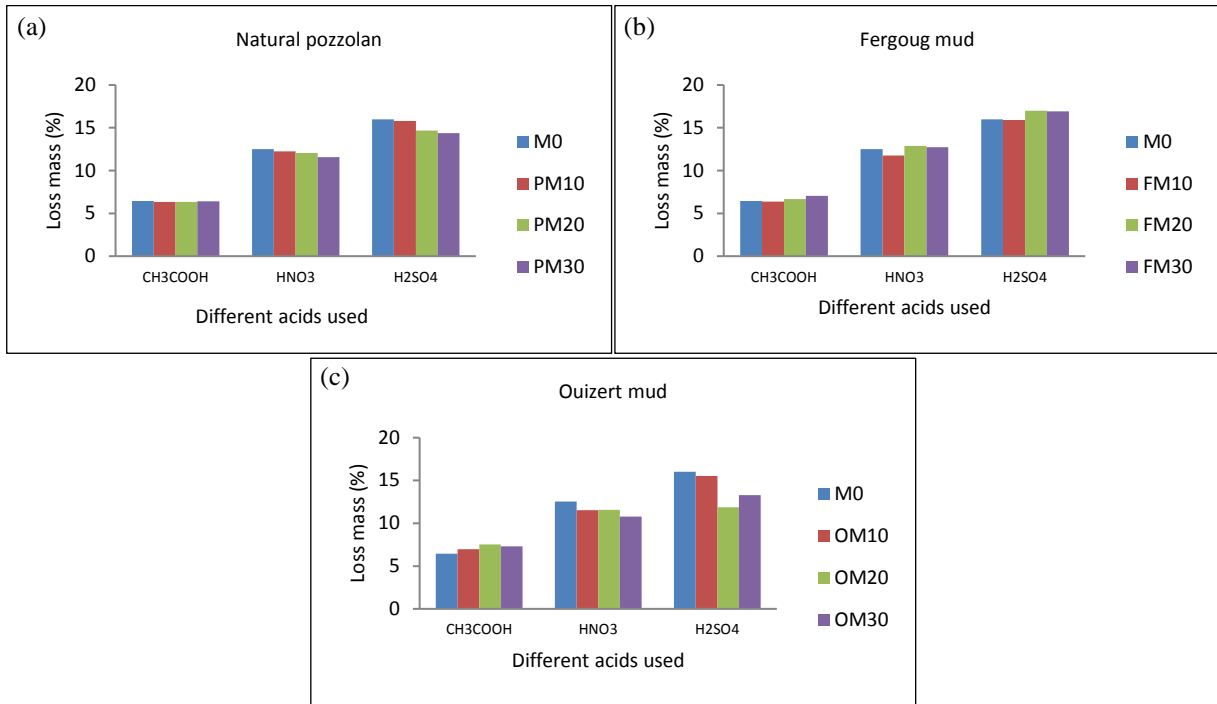


Fig. 15- Comparison of the mass loss of the various mortars to acids attacks at 28 days.

3.4.1.5 Visual state of the test pieces after acid attacks

It is possible to determine the degree of degradation of the specimens from the visual state of these specimens after the acid attacks, as shown in figure 16. After 28 days, it can easily be seen that the specimens immersed in sulfuric acid are the most degraded; next come those in nitric acid.



Fig. 16- State of the specimens after immersion for 28 days in the various acids.

3.4.2 Water absorption test

The pores formed are capillary pores because they are not visible to the naked eye. The measurement of absorption is a simple and easy way to characterize the absorption kinetics of materials. The higher the sorptivity, the more rapidly the material can be invaded by the liquid in contact. The sorptivity is also a property that characterizes the disposition of pores within the material to absorb water and transmit it by capillarity. It is defined by the following equation:

$$W(\%) = \frac{(Mh - Ms)}{Ms} \times 100 \tag{2}$$

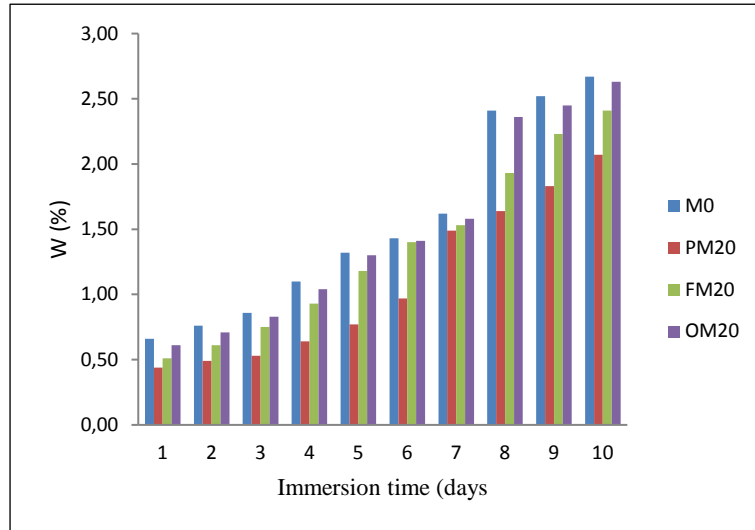
Mh: The mass of the sample after absorption of water,

Ms: The mass of the dry sample.

Capillary absorption tells us how high water can rise through the open porosity of the concrete; this open porosity is produced by the surface tension of the liquid. Figure 17 indicates that all natural and artificial pozzolanic mortars are

beneficial for the absorption test because they have a smaller absorption as compared to that of the control mortar, at all maturities. Mortar with 20% natural pozzolan (PM20) gave the smallest absorption, followed by the mortar with 20% of calcined mud from Fergoug dam (FM20), and finally comes the mortar with 20% of calcined mud from Ouizert dam (OM20).

The preceding results allow us to say that the incorporation of natural and artificial pozzolans reduces the volume of capillary pores by filling them with the second-generation C-S-H gel resulting from the silica of pozzolan and portlandite ( $\text{Ca}(\text{OH})_2$ ), a hydrate produced by the hydration of cement, [11]; [20].



*Fig. 17- Evolution of the absorption of different mortars.*

### 3.4.3 Monitoring the pH values of the acid solution

The reading of the pH of the acid solution was monitored by means of a pH meter during the entire immersion period of the test pieces, figure 18.

The results obtained are presented in the form of graphs in figure 19. This representation allows us to determine to what extent the sample (PM10) was attacked by the acid solution in order to show the impact of adding natural pozzolan on the resistance of mortars to the various acids used. There was an increase in the pH value for all acids over the nine-day follow-up period. The pH increases over the 9-day immersion period were 0.45, 0.74 and 2.15 units for acetic, nitric and sulfuric acids, respectively. It can therefore be said that the sulfuric has the most significant attack.



*Fig. 18- Apparatus for reading the pH of the acid solution.*

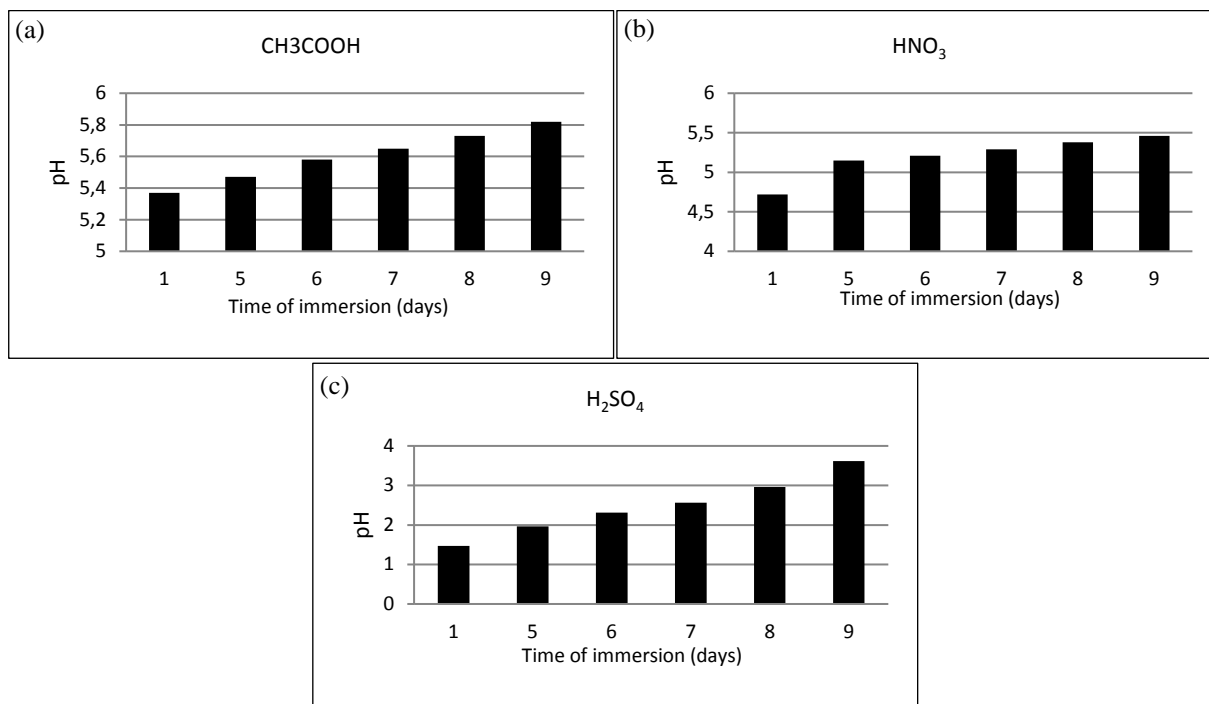


Fig. 19- Monitoring of the pH reading of the different acid solutions.

#### 4 Conclusion

The research presented in this study is part of the valorization of the sediments resulting from the dredging of the dams of Fergoug and Ouizert on the one hand and the natural pozzolana from the town of Beni-Saf on the other. Our contribution is essentially oriented towards the eventual utilization of these pozzolans in the formulation of concrete and mortar for the needs of the *building* and construction sector. These materials are considered as mineral raw materials which represent an appreciable potential for construction in view of their considerable availability in Algeria. The dam of Fergoug, for example, which is silted up by more than 90%, has become a major concern for many of us. On the other hand, the physical and chemical characterization of these materials has shown their suitability for use as pozzolanic materials. This is the main reason that has pushed us to develop this pozzolanic aspect through the different tests carried out in this study. The tests carried out during this work led to the following results:

- The incorporation of mineral additions significantly affects the workability of fresh mortars. Indeed, all additions used (natural pozzolana and calcined mud) increase the workability of the mortar, thus making the mixture more plastic.
- Testing the compressive strength of mortars proved that the mechanical performance of the pozzolanic mortars is better than the strength of the control mortar. The mud from Fergoug dam with a substitution rate of 10% (FM10) gives the best mechanical performance,
- The tests evidenced the reliability of mud calcination because the mortars formulated with mud calcined at 850 °C/hour showed performances which are close to those of the control mortar,
- The restrained shrinkage test, where the visual assessment is critical, revealed that shrinkage and swelling on the different mortars made with natural and artificial pozzolans are less than those observed on the control mortar,
- The incorporation of natural pozzolana improves the resistance of mortars to the attack of all the acids used. The inclusion of calcined mud from the dam of Ouizert improves the resistance of mortars to the attack of nitric and sulfuric acids. The incorporation of calcined mud from Fergoug dam makes these mortars more resistant to the different acids. The strengths of these mortars are close to those of the control mortar. It was found that sulfuric acid was the most aggressive, then comes the nitric acid, and finally the acetic acid,
- The visual examination of mortar specimens made it possible to highlight the state of each mortar before and after the attack of the acids (H<sub>2</sub>SO<sub>4</sub>, CH<sub>3</sub>COOH and HNO<sub>3</sub>). The sulfuric acid attack was the most severe,

- All natural and artificial pozzolanic mortars have been found to be beneficial for the absorption test because they have a smaller absorption than that of the control mortar, at all maturities. The mortar with 20% natural pozzolana (PM20) exhibited the smallest absorption,
- The follow-up observations of the pH confirmed that the pozzolanic mortars resist well to the acid solutions used.

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