# Ecological and economical mortars made with dune sand and cements in combination with local mineral additions

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**Abstract:** The aim of this study is to formulate and characterize, with different methods, mortars prepared with dune sand and Algerian clinker in combination with several admixtures, natural additions or industrial by-products as pozzolan, granulated blast furnace slag and marble powder. Mineralogical and chemical compositions of the initial products were studied with X-ray diffraction (XRD), SEM, EDX and X fluorescence (XRF). Grain size distribution of anhydrous cements and additions (absolute density, Blaine specific surface (SSB) and laser granulometry) were carried out. Mortar strengths are acceptable at 28 days: between 5 and 6 MPa in bending test and between 28 and 41 MPa in compressive test. These resistances improve clearly to reach high performances after one year: between 7 and 9 MPa in flexural test and between 43 and 61 MPa in compressive test. The microstructure was studied using SEM coupled with microanalysis EDX; these observations explained the increase mechanical strength. Results showed that it is possible to obtain mortars with high mechanical performances in long-term and, ecological and especially economic advantages with a good formulation of those mortars containing dune sand and additive cements (binary, ternary or quaternary) with super plasticizer adjuvant.

**Keywords:** mix design, characterization, pozzolan, slag, marble, dune sand Algeria

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## 1 Introduction

The Algerian South consists of an inexhaustible dune sand reserve, it is very important to investigate possibilities to insert this local material in mortars. In the last decades, the studies were multiplying on the physico-chemical characteristics of this sand characterized by its smoothness due to wind carriage and his very thin granular extend (Bédérina et al., 2005).

On the other hand, most cement plants consume a lot of fossil energy and produce a large amount of undesirable products, which affect the environment. In order to reduce energy consumption and $CO_2$ emission and increase production, cement manufacturers are blending or intergrinding mineral additions such as slag, natural pozzolan, sand and limestone (Kenai, Soboyejo and Soboyejo, 2004). The valorization of waste and industrial by-products in civil engineering can give encouraging results as well in terms of economy, ecology and behavior (Marchal, 2002).

The additions of the blast furnace slag to cement present an economical interest in cement industry and a technical interest in the field of construction (Samet and Chaabouni, 2003; Pal, Mukherjee and Pathak, 2003). The energy cost of this by-product is practically zero; nevertheless it requires more energy for crushing than the clinker (Dubosc, 1998).

The increase of the specific surface (3,500 $cm^2/g$ to 4,200 $cm^2/g$) clearly improves the mechanical resistances for the concrete containing up to 30% of slag

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(comparable resistances to those of concrete without slag) (Naceri and Messaoudenne, 2006).

The substitute of ordinary cement by the pozzolan of Beni-Saf (Algeria), available in considerable quantities, makes it possible to improve mechanical strengths of concrete at medium and later ages. Former American studies on the use of pozzolan indicate that cements mixed with pozzolan have damp-proof qualities (San Francisco Golden Gate). Algerian pozzolan (Beni Saf) was previously studied (Mebrouki et al., 2004; Kaid et al., 2009).

Chaid et al. (2004) studied the addition of ultrafine pozzolan in the aim to obtain high performances concretes. He obtained at 28 days compressive strength of concretes higher than 50 MPa with the help of super plasticizer and correctly adjusted composition.

Recently, the use of the marble powder as a material of replacement was studied. Agarwal and Gulati (2006) showed that the presence of marble powder in the matrix improved the compressive strength at the young age. Topçu, Bilir and Uygunoglu (2009) pointed out the filler effect of marble powder: the workability of self compacting concretes was not affected but mechanical decrease for high marble powder content (200 kg/m$^3$). According to other authors (Kavas and Olgun, 2008), short and long-term strength of mortars can be improved by incorporation of slag and fly-ashes in the matrix.

Combination of several cementing additions (2 or 3) with clinker and gypsum allows the development of new types of hydraulic binders (ternary or quaternary cements) having higher mechanical properties and better durability than the Portland cement CEM I (clinker alone) (Laldji, Phithaksounthone and Tagnit-Hamou, 2004).

In this experimental work, we studied the influence of cements in combination with several admixtures, natural additions or industrial by-products on the mechanical performances of the mortars containing dune sand.

## 2 Products used

### 2.1 Clinker

In this study, clinker (CLK) of the cement factory of Ain-El-Kébira (Sétif- Algeria) was used as activator, especially for blast furnace slag and natural volcanic pouzzolan ash. Its mineralogical composition (Bogue composition) is presented in Table 1. The natural gypsum (G) is used as regulator of setting time. In comparison with classical clinker, this one is rich in $C_2S$ and $C_4AF$.

**Table 1 Clinker Bogue composition (%, by weight)**

| $C_3S$ | $\beta C_2S$ | $C_3A$ | $C_4AF$ |
|---|---|---|---|
| 61.23 | 17.13 | 3.57 | 15.60 |

### 2.2 Granulated slag

The granulated blast furnace slag (GBFS) used in this study was an industrial by-product from iron and steel industry obtained from the metallurgic unit of EL-Hadjar (Annaba-Algeria). This slag was studied by Achoura (2005) as sand and as filler.

### 2.3 Pozzolan

Natural pozzolan (PZ) extracted from Beni-Saf quarry, in the west of Algeria, has the appearance of crushed pumice stone and slag. This material was described by Belas Belaribi, Semcha and Laoufi, (2003). Durability of mortar with Beni Saf pozzolan was studied by Kaid *et al.* (2009) and performance characteristics of lightweight aggregate concrete containing this pozzolan by Mouli and Khelafi (2008) and Ghrici, Kenai and Meziane (2006).

### 2.4 Marble powder

The marble powder used (MP) was a waste of flooring tile from industry (Bordj Bou Arréridj, Algeria).

### 2.5 Chemical admixture

A super plasticizer (SP) provided by the company GRANITEX (Algiers, Algeria) was used. This super plasticizer, MEDAFLUID SFR122, contains combined polymers. It is recommended for the production of the rheoplastic concretes with a very low E/C ratio. It allows the increase of the mechanical resistances in the long term. It is applicable to any type of cements which is conforming to the standard EN 934-2. The interval of recommended use is between 0.8% and 2.5% of the weight of the binder. The set of concrete is obtained in a good delay with 1% of additive. Its physical and chemical characteristics are presented in Table 2.

**Table 2 Physical and chemical characteristics of the additive**

| Nature | Apparent density | pH (1% in water) | $Cl^-$ content | Dry extract |
|---|---|---|---|---|
| Liquid | 1.20± 0.02 | 6.6 ± 0.1 | <1 g/L | 40% |

## 3 Mineral and chemical analysis

### 3.1 X-ray diffraction analysis

The mineralogical composition was determined by X-ray diffraction. The XRD data were collected with Philips PW 3710 X-ray diffractometer with Bragg–Brentano geometry using Ni-filtered Cu Kα radiation, operating with the voltage of 30 kV and emission current of 20 mA. The step-scan covered the angular range 2–60° (2*θ*) in steps of 2*θ* = 0.02°.

The results of Figures 1, 2, 3, 4 and 5 highlight:

– for the CLK: prevalence of alite ($C_3S$) and the absence of alkali,

– for G: composed quasi entirely of gypsum ($CaSO_4.2H_2O$) with quartz traces ($SiO_2$),

– for the GBFS: amorphous material (vitrified),

– for the PZ: prevalence of cordierite ($2MgO.2Al_2O_3.5SiO_2$) and analcime ($Al_2O_3.Na_2O.4SiO_2.2H_2O$), small quantities of hematite ($Fe_2O_3$) and cristobalite ($SiO_2$).

– and for the MP: composed only of calcite ($CaCO_3$).

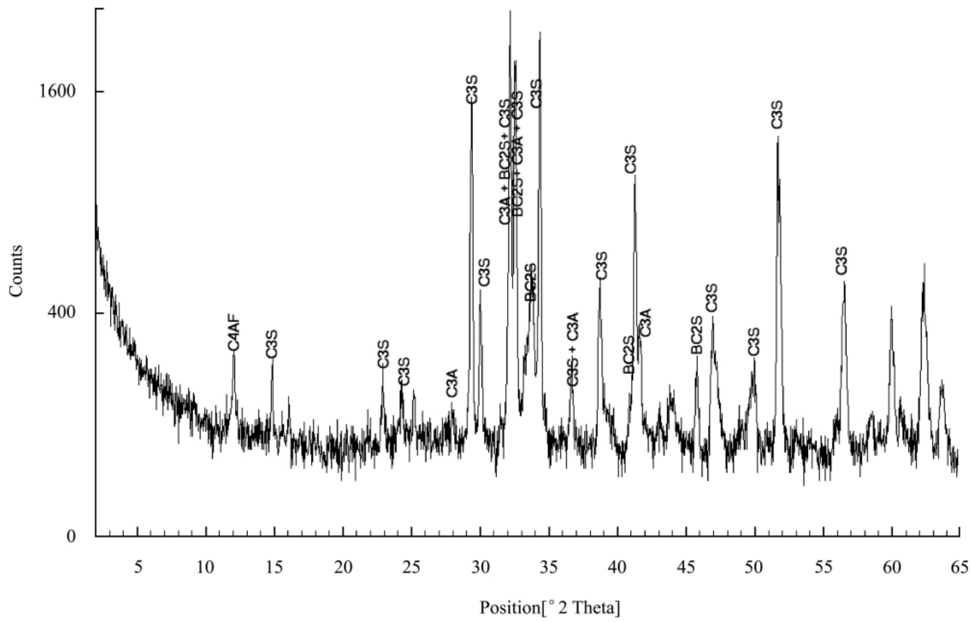



Figure 1 X-ray diffraction of the clinker (Cu Kα radiation, Ni filtered)

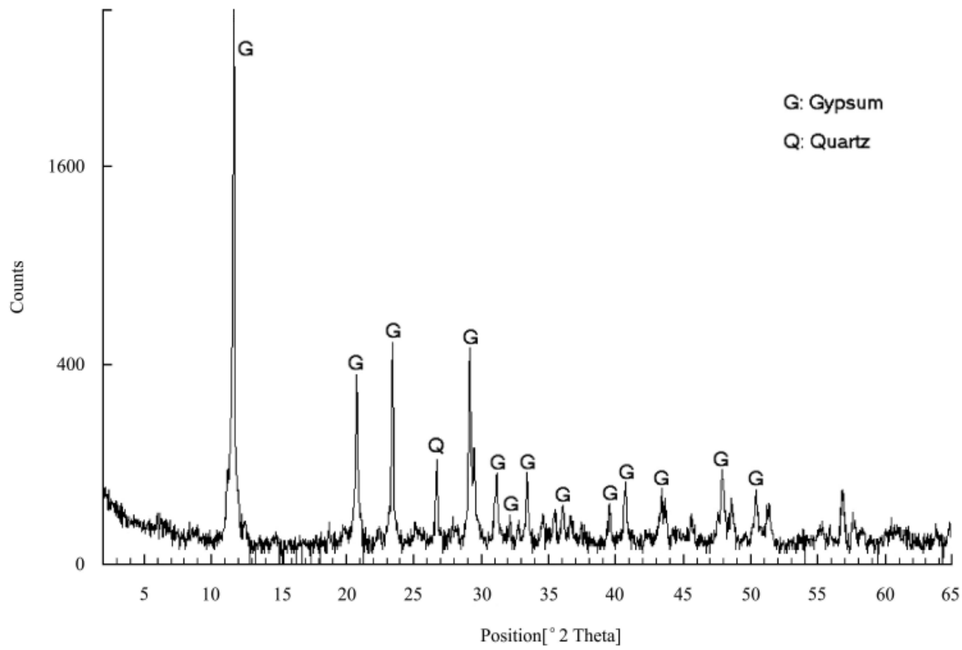



Figure 2 X-ray diffraction of the gypsum (Cu Kα radiation, Ni filtered)

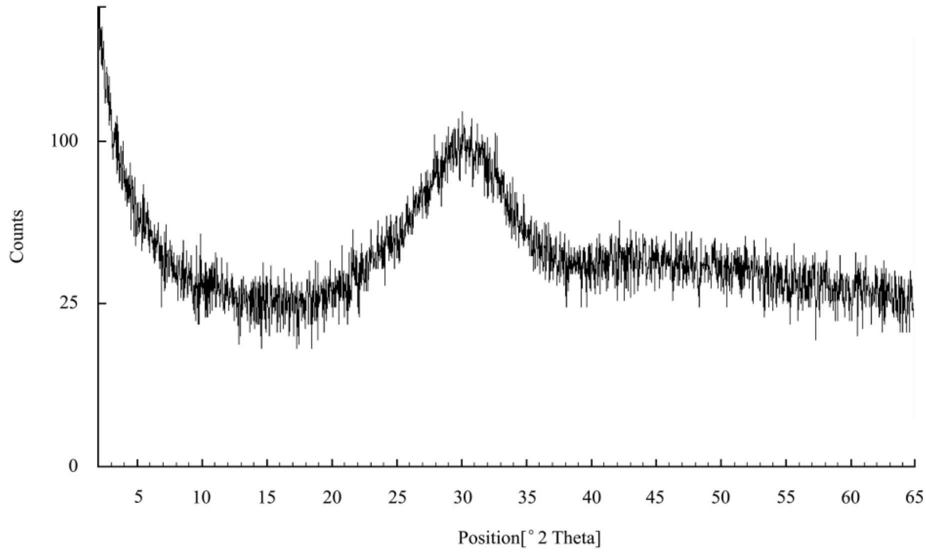



Figure 3 X-ray diffraction of the GBFS (Cu Kα radiation, Ni filtered)

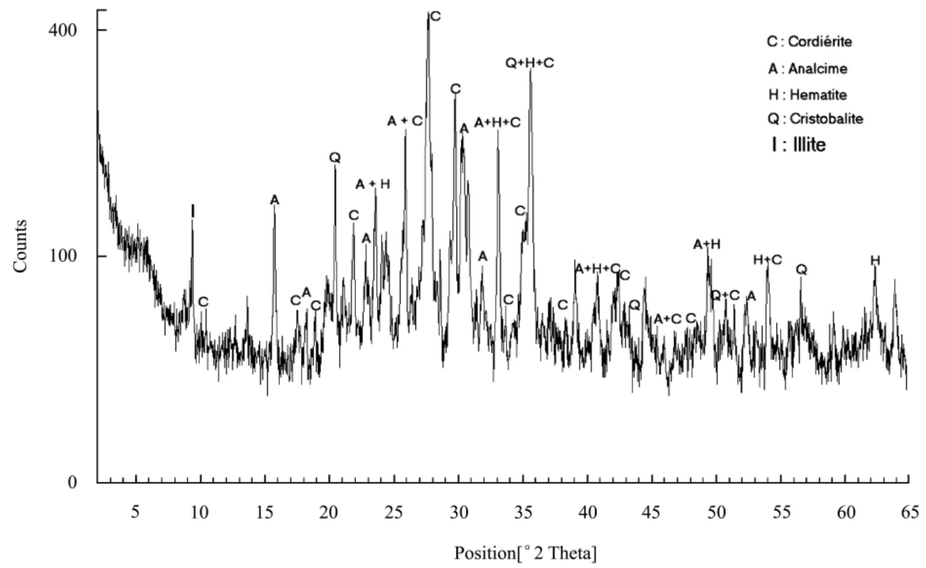



Figure 4 X-ray diffraction of the pozzolan (Cu Kα radiation, Ni filtered)

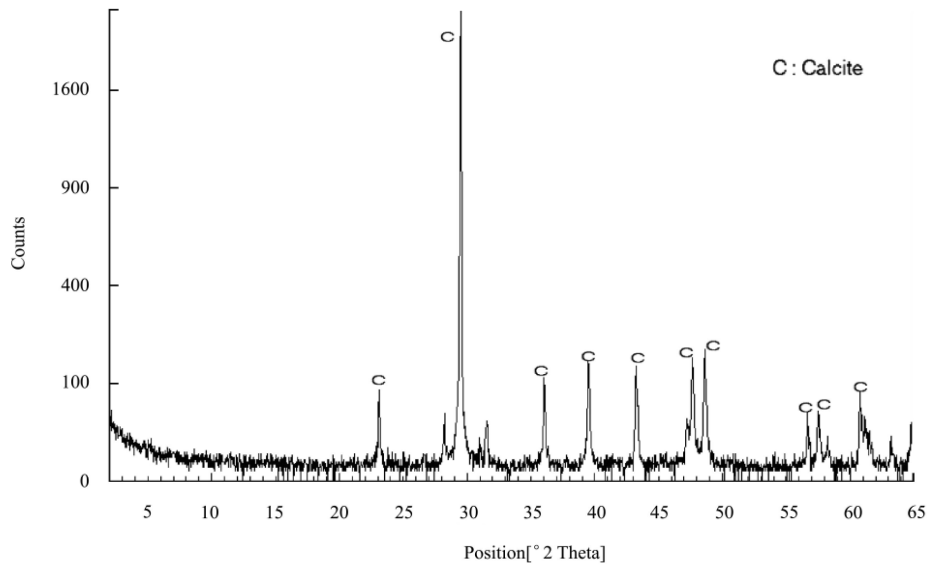
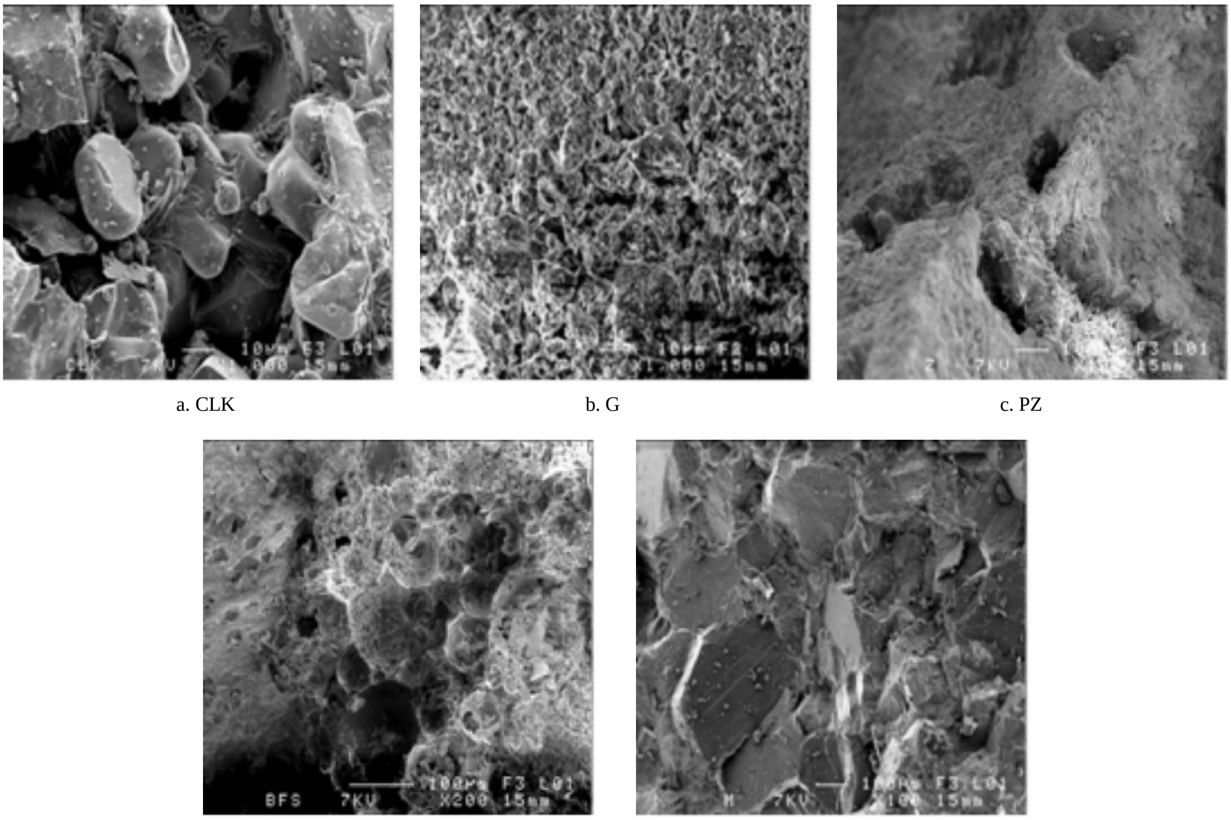



Figure 5 X-ray diffraction of the marble (Cu Kα radiation, Ni filtered)

### 3.2 SEM observations

SEM observations were conducted on a SEM FED (JEOL-JSM-6301F) using an accelerating voltage of 7 kV and a working distance of 15 mm. All the initial products were observed before crushing (Figure 6).



a. CLK b. G c. PZ

d. GBFS e. MP

Figure 6 SEM observation of materials

Micro analysis data were collected using conventional JEOL-JSM-6400 Scanning microscope. Micro analysis of the samples was performed using an accelerating voltage of 20 kV and a working distance of 39 mm (Table 3).

These photos indicate:

1) for the CLK: particles of a few tens of micron, adherent ones with the others with a great porosity coming from hardening,

2) for G: its saccharoid form,

3) the PZ is presented in the form of ash and slag,

4) for the GBFS: a granulated form soaked with a closed porosity and finally for

5) the marble: poly crystalline with well formed crystals tangled up one in another.

The microanalysis by EDX confirms the observations of the analysis by diffraction.

Elementary proportioning of the CLK, the PZ and the GBFS are carried back in Table 3.

**Table 3 Chemical composition of the clinker, pozzolan and slag**

| | Clinker | | Pozzolan | | Blast furnace slag | |
|---|---|---|---|---|---|---|
| | % mass | % atom | % mass | % atom | % mass | % atom |
| O | 35.57 | 56.53 | 54.16 | 70.11 | 47.39 | 65.38 |
| Mg | 0.80 | 0.83 | 1.75 | 1.49 | 2.65 | 2.40 |
| Al | 0.92 | 0.87 | 7.78 | 5.97 | 3.76 | 3.07 |
| Si | 8.97 | 8.12 | 19.00 | 14.01 | 16.91 | 13.29 |
| K | 0.65 | 0.42 | 0.87 | 0.46 | 0.73 | 0.41 |
| Ca | 50.50 | 32.04 | 8.66 | 4.47 | 25.19 | 13.87 |
| Fe | 2.59 | 1.18 | 5.76 | 2.14 | | |
| Ti | | | 1.01 | 0.44 | | |
| Na | | | 1.01 | 0.91 | | |
| Mn | | | | | 2.61 | 1.05 |
| S | | | | | 0.76 | 0.52 |

From these analyses, in one hand, it can be observed that the most calcareous is the clinker. The slag contains half less Ca than clinker and the pozzolan 1/6th. In the other hand, the Si content increased as the Ca content decreased. During the hydration process, the clinker would release lime. This lime would be consumed by the slag and the pozzolan.

## 4 Experimental procedures

### 4.1 Preparation of mixtures

The products were crushed to obtain a granulometry lower than 1mm before being carefully mixed, then quadded and prepared according to the proportions given in Table 4. Following the NF EN 197-1 standard, the binary cement (BC) is similar to a CEM I/B-S, and the four ternary cement (TC) and the quaternary cement (QC) are similar to CEMII/B-M. The grinding of the six types of cements were performed in vibratory mills. The chemical composition of cements thus prepared was determined by X-ray fluorescence (Table 5).

**Table 4 Mixes composition of binders (%mass)**

| Binders | Clinker | Gypsum | Slag | Pozzolan | Marble | |
|---|---|---|---|---|---|---|
| BC | 65 | 5 | 30 | 0 | 0 | Binary cement |
| TC1 | 65 | 5 | 25 | 0 | 5 | Ternary cement 1 |
| TC2 | 65 | 5 | 20 | 0 | 10 | Ternary cement 2 |
| TC3 | 65 | 5 | 15 | 0 | 15 | Ternary cement 3 |
| TC4 | 65 | 5 | 0 | 22 | 8 | Ternary cement 4 |
| QC | 65 | 5 | 11 | 11 | 8 | Quaternary cement |

**Table 5 Chemical composition by X fluorescence (%mass)**

| Binders | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $K_2O$ | $Na_2O$ | $Cl^-$ |
|---|---|---|---|---|---|---|---|---|---|
| BC | 24.87 | 5.33 | 4.77 | 58.81 | 2.36 | 2.20 | 0.36 | 0.06 | 0.00 |
| TC1 | 23.03 | 4.85 | 3.93 | 58.16 | 2.22 | 2.79 | 0.40 | 0.13 | 0.02 |
| TC2 | 20.75 | 4.38 | 3.69 | 57.30 | 2.35 | 2.29 | 0.37 | 0.10 | 0.04 |
| TC3 | 18.95 | 4.00 | 3.55 | 58.28 | 2.25 | 2.57 | 0.33 | 0.14 | 0.06 |
| TC4 | 23.64 | 6.39 | 5.38 | 52.01 | 2.31 | 2.48 | 0.53 | 0.21 | 0.04 |
| QC | 22.48 | 5.45 | 4.96 | 57.59 | 2.30 | 1.49 | 0.34 | 0.13 | 0.03 |

The absolute density of cements and Blaine specific surface (BSS) are carried back in Table 6.

**Table 6 Absolute density and specific surface Blaine**

| Binders | BC | TC1 | TC2 | TC3 | TC4 | QC |
|---|---|---|---|---|---|---|
| Absolute density | 3.14 | 3.11 | 3.09 | 3.04 | 3.01 | 3.13 |
| BSS/$cm^2 \cdot g^{-1}$ | 4,170 | 4,581 | 4,770 | 4,532 | 4,438 | 4,392 |

We can observe that for BC, TC1, TC2, and TC3, more the addition of marble powder increases (or more the addition of slag decrease), more the density of cements decreases. It can be due to high density of slag and lower density of marble. On the contrary the densities of binary and quaternary cements are practically identical.

Granulometric analysis data given by a laser particle-measurement instrument were collected with CILAS 2 equipment.

The granulometry of the binders (Figure 7) are practically the same for all types of cement. It is lower than 70 μm, with a maximum frequency around 11 μm.

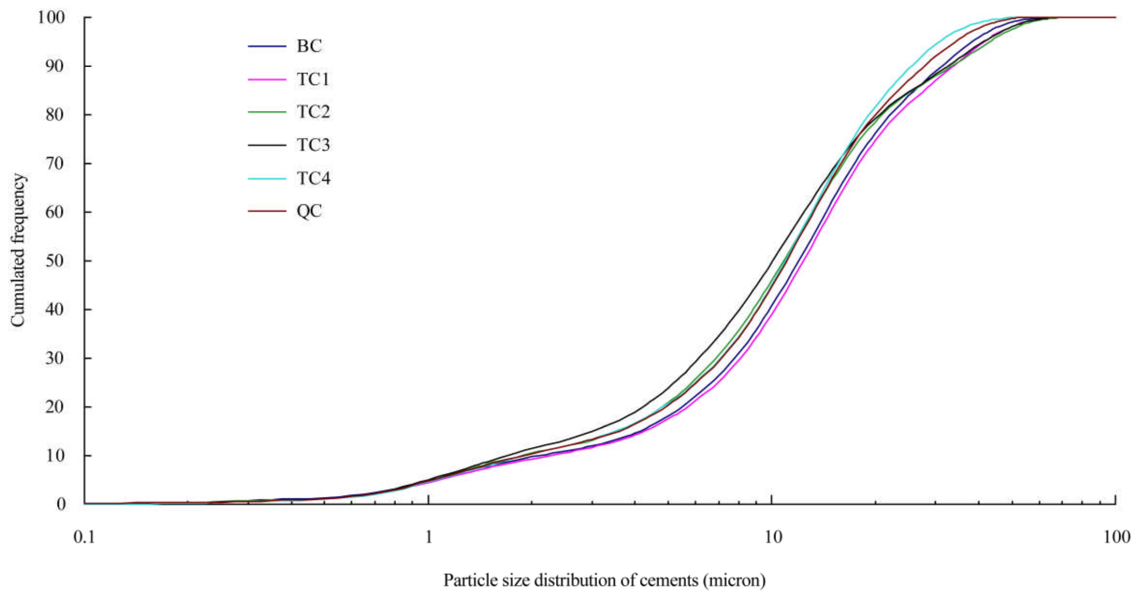



Figure 7 Granulometry of the binders

### 4.2 Characterization of the dune sand

The dune sand (DS) was taken from "Maîter wadi", located between the hills of Boussaâda. This fine grained sand was carried by southerly winds. According to standard NF P18-304, the granulometric study was carried out after washing in a sieve of 80 µm and stored in the drying oven during 24 h.

Figure 8 shows that 73% of the grains have a diameter lower than 0.63 mm. The fineness modulus is worth 2.39 (NF P 18-560). The visual sand equivalent is about 95% and 75% by piston; the methylene blue value is about 0.1. Therefore, we can conclude that it is clean sand. Its measured specific gravity is about 2.6.

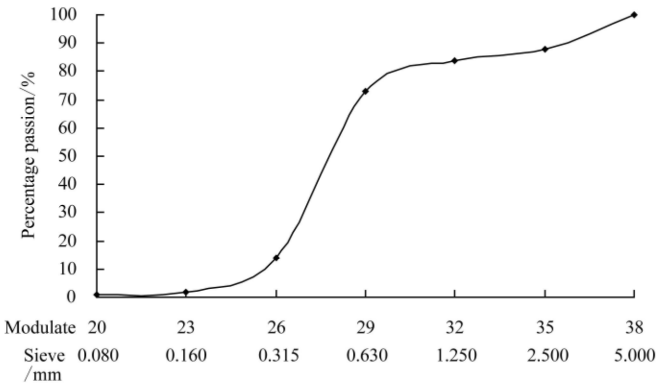



Figure 8 Granulometry of the dune sand

The mineralogical composition (Figure 9) determined by X-rays diffraction shows that this sand was siliceous, more than 95% of quartz and some traces of calcite.

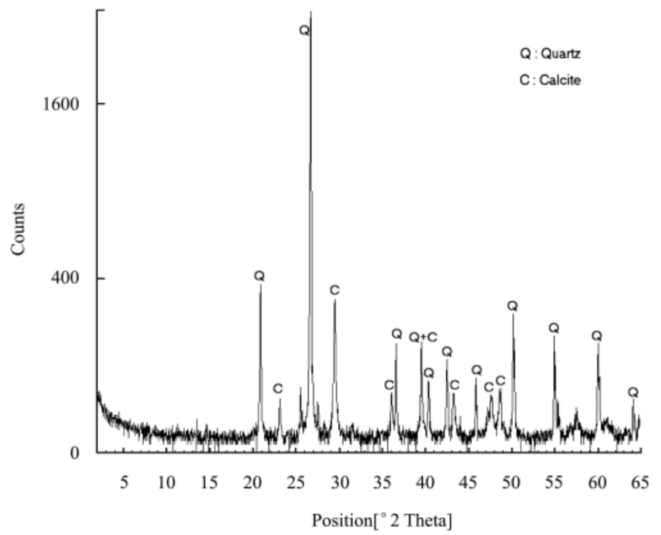



Figure 9 X-ray diffraction of the dune sand (Cu Kα radiation, Ni filtered)

### 4.3 Formulation of the mortars and mechanical characterization

Dune sand, cement and the chemical admixture were mixed with water according to the proportions presented in Table 7, with a water/binder ratio = 0.45. After production of mortars, the moulded specimens were covered with plastics sheets at (20±1)℃ for (24±1) h in accordance with the standard EN 196 1. The samples were transferred and stored in water until the time of testing. For each mix, prisms of size 4 × 4 × 16 $cm^3$ were tested to determine flexural and compressive strengths, respectively, at 7, 28 days and 1 year. The results are presented in Figures 10 and 11.

**Table 7 Composition of the mortar**

| Binder/g | DS/g | Water/g | SP 1%/mL |
|---|---|---|---|
| 450 | 1,350 | 202.5 | 4 |

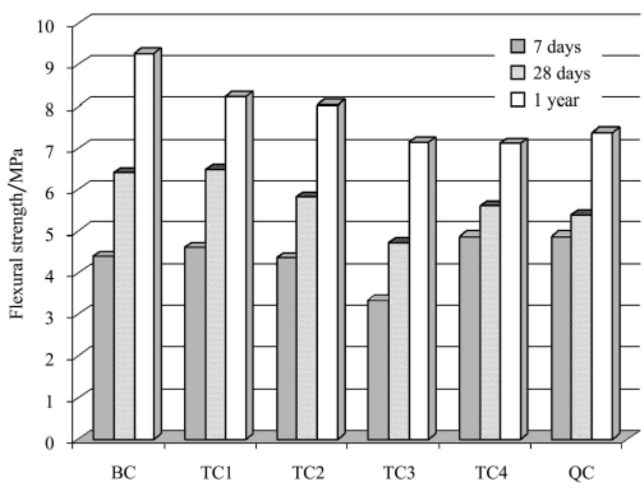



Figure 10 Evolution of flexural strengths

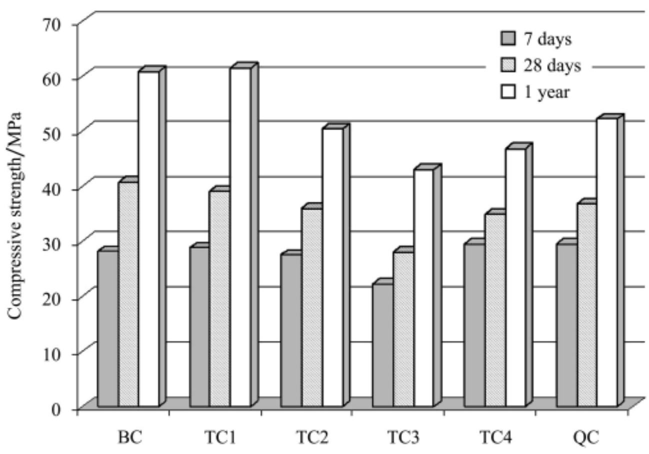



Figure 11 Evolution of compressive strengths

The mortar strengths are acceptable at 28 days: between 5 to 6 MPa in flexural test and between 28 to

41 MPa in compressive test. These resistances improve clearly to reach very important performances after 1 year between 7 to 9 MPa in flexural test and between 43 to 61 MPa in compressive test (strengths improvement about 50% between 28 days and one year). Highest strengths are obtained with slag addition (30%) BC. 5% replacement of slag by 5% of marble conduct to the same results (TC1 cement). Beyond 10% replacement of slag by marble (TC2 and TC3), an important decrease of strength (especially of compressive strength) could be observed. Contrary to popular belief, the mortar with pozzolan (TC4 and QC) gives the highest strength at short term. The quaternary cement (QC) gives similar strength at short term and quite correct strength at 28 days (equivalent CEM II32.5) and at a long term (51 MPa).

The evolution of resistances according to time is explained by the latent hydraulic property of the hydration of the mineral additions (slag and pozzolan).

### 4.4 Internal microstructure

The study of the microstructure, carried out by SEM and confirmed by microanalysis showed that after 28 days and one year, the hydration of the cementing matrix led to the formation of CSH (calcium silicate hydrate-gel-like flocks), small quantities of portlandite crystals CH and the ettringitte (fine needle-like crystals) $C_3A$, $3CaSO_4$, $32H_2O$ ( Figure 12 and 13).

We observe a less porous structure, explained by the specific surface of cement greater than 4,100 $cm^2/g$, the addition of the industrial fillers (slag and marble powder) and the use of adjuvant. Whether with blast furnace slag or with Beni Saf pozzolan to a lesser extent, internal porosity observed by SEM is very low. The amount of portlandite is also low: in the both cases, it reacts to form a second generation of silicates and aluminates hydrated lime. These reactions conduct to a sealing of the porosity.

We can observe a very tight contact between marble grains and cement hydrates but the marble powder seems to be non reactive (no epithaxy between hydrates and $CaCO_3$). The marble grains are known to be nucleation sites at the beginning of the hydration process and contributes to block the porosity.

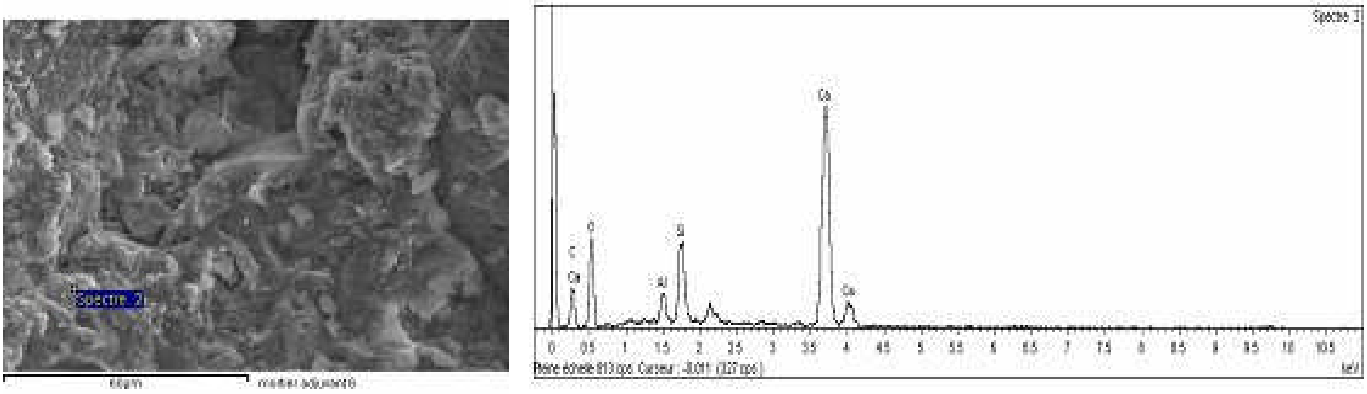



Figure 12 SEM observation of mortar containing quaternary cement (QC)

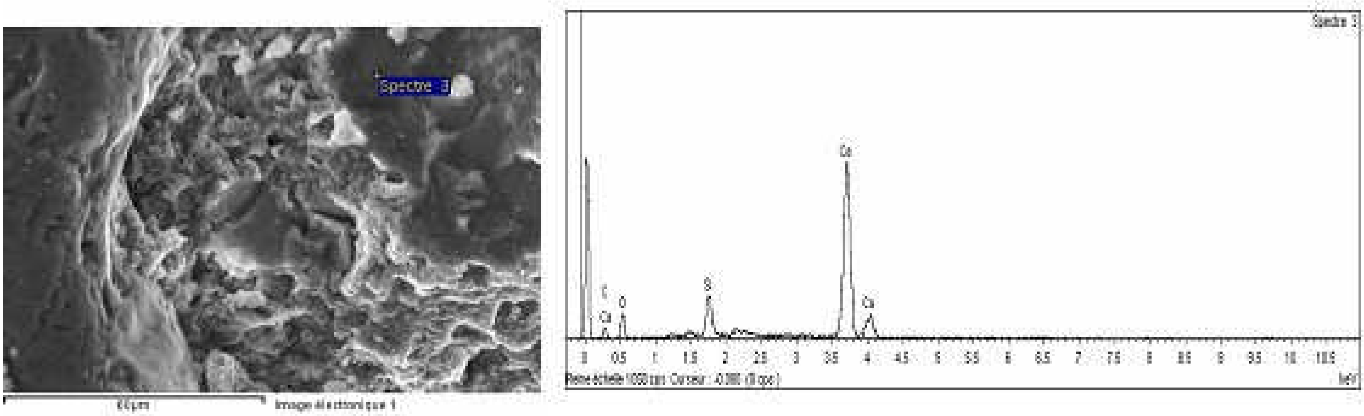



Figure 13 SEM observation of mortar containing ternary cement (TC1)

## 5 Conclusions

Results showed that it was possible to obtain mortars with dune sand and local by-products: blast furnace slag, natural pozzolans (Beni Saf volcanic ashes) and marble powder.

These mortars are very economical due to the used of low cost materials: the volcanic pozzolan and marble powder are natural materials that only need a low energy grinding; the slag is a by-product from the steel manufacturing industry that needs a grinding with higher energy than the previous two. These three fillers do not need any calorific energy compared to clinker manufacturing.

From an environmental perspective, no more carbon dioxide is produced when these three materials are used. Furthermore, slag and powder marble are waste that should be stocked in adapted landfills.

The dune sand, an inexhaustible reserve in the north African country, exhibits a very thin granulometry, that is not recommended for concrete mixes, but it presents a high content of quartz without argilous particle.

The proposed mortar compositions give acceptable strength at short and long terms. The highest strength is obtained with binary cement containing 65% clinker, 30% slag and 5% gypsum. The replacement (up to 10%) of slag by marble powder conducts to an acceptable decrease of strength. The slag presents a latent hydraulicity induced by clinker hydration. The use of volcanic pozzolan gives to lower strength at long term but to higher strength at short term. Volcanic pozzolan are not hydraulic but reacted with lime released by clinker hydration (pozzolanic reaction). High short term strength can be due to the high reactivity of this pozzolan: pseudo-crystallized or amorphous siliceous components observed by X-ray diffraction.

SEM observations indicate a very low porosity. The initial porosity is low due to the low water to cement ratio and marble powder content. In a second time, the porosity is blocked by secondary hydrates (slag – lime reaction and pozzolan – lime reaction).

The quaternary cement proposed is a good compromise: it gives high short term strength, normal strength at 28 days (similar to a CEMII 32,5) and good strength after one year.

Proposed cement with low porosity and low $Ca(OH)_2$ content should be resistant face to biological, physical and chemical attacks.

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