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# EFFECT OF THE SETTING AGENT (LIME) ON THE PHYSICO-CHEMICAL PROPERTIES OF SLAG CEMENT AND MECHANICAL BEHAVIOR OF MORTAR

A. Naceri* and M.S. Bouglada
*Laboratory of Materials, Department of Civil Engineering*
*Faculty of Sciences and Engineering, University of M'sila, Algeria*

## Abstract

The increase of the pozzolanic admixture (slag) added as a replacement for cement causes a disadvantage of a longer initial setting time and a slower strength development in cold time. The aim of the present experimental work is to evaluate the influence of mineral activator (hydrated lime) of slag cement (latent hydraulicity) on the physico-chemical properties of slag cement (C.E.M II) and the mechanical behavior (Flexural and compressive strengths) for the mortar. The activation method by hydrated lime of slag cement activates the hydration process, influenced the size of particles and leads to the formation of ettringite and CSH (tobermorite) at the early ages of hydration. In this experimental study, the setting agent $Ca(OH)_2$ used for activation slag cement is used in the proportions of 0%, 2%, 4%, 6%, 8% and 10% by various methods (substitution and addition by mass of slag cement). The physical properties of cements (C.E.M II) activated by the calcium hydroxide at anhydrous state and the state hydrated (specific weight, fineness, consistency of the cement pastes and setting times) thus the characteristics of the mortars made at their bases, such as, the mechanical behavior (Flexural and compressive strengths for the mortar) after 7, 28 and 90 days were studied.

**Keywords:** Activation method; slag; lime; cement, mortar; mechanical strength



## 1. Introduction

The Portland cement (C.E.M II) with pozzolanic admixture (slag) presents a hardening slowed down at its initial period in comparison with an ordinary Portland cement (cement without secondary component : C.E.M I) [1,2]. This latent property of cement with mineral addition (C.E.M II), requires the use of a efficace activation, chemical, mechanical or thermal [3].

The blended cements with mineral additions (C.E.M II) have a latent setting times than

*E-mail address of the corresponding author: abdelghani_naceri@yahoo.fr (A. Naceri)

ordinary Portlands cements (C.E.M I), especially in the case of concreting in cold time [4]. It is known that setting times can be shortened [5]:

- by high fineness (specific surface) of cement,
- or by the use of accelerating admixtures (NaOH, KOH,......).

The Portland cement (C.E.M II) with pozzolanic admixture (slag) is low in $C_3S$ (tricalcium silicate), low in $C_3A$ (tricalcium aluminate) and low heat of hydration and high long term strength.

Hydrated lime (calcium hydroxide) or quicklime (calcium oxide) is commonly used in building construction. Lime is obtained from calcium decarbonation above 900 °C. The lime may be quicklime or slacked lime after to have been hydrated air calcium lime is sometimes called fat lime [6]. Air lime may come in various forms : rock or powder for quicklime, powder, suspension in water (milk of lime) or lime putty for hydrated lime. The lime is used in industry thanks to its numerous physico-chemical properties (basicity, specific area, etc.). Lime is mainly used in construction (masonry mortar), water treatment (domestic, industrial or waste water treatment), soil treatment, etc ., because of its neutralising capacity (chemical base).

The lime saturation factor (LSF) controls the potential $C_3S$ to $C_2S$ ratio in the finished cement. $C_3S$ governs the early age strength development while $C_2S$ governs the later age strength.

The activation of the slag cement by fine lime (lime hydroxide) is a simple and economic method especially for cement containing not very reactive slag (low coefficient of activity) [7].

Lime plays 2 roles [8]:

- to hydrate the slag of granulated blast furnace (formation of the hydrated compounds CSH : tobermorite),
- to maintain cement with a pH > 12,
- the hydration of the clinker (formation of ettringite) accelerates.

The objective of this present experimental work is to evaluate the influence of mineral activator (hydrated lime) of slag cement (latent hydraulicity) on the physico-chemical properties of slag cement (C.E.M II) and the mechanical behavior (Flexural and compressive strengths) for the mortar.

## 2. Characteristics of Used Materials

### *2.1 Natural sand (fine aggregates)*

The fineness modulus calculated was $M_f$ = 1,76. The information on the physical properties of the natural sand used is given in Table 1. Its chemical composition is shown in Table 2.

Table 1. Characteristics of dune sand used in the tests

| Materials | Absolute density | Apparent density (Kg/l) | Compactness (%) | Porosity (%) | Sand equivalent value (sight/test) |
|---|---|---|---|---|---|
| Dune sand | 2.56 | 1.64 | 64.06 | 35.94 | 76.78 |

Table 2. Chemical composition (%, by weight) of sand dune used

| Oxides % | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $CaO_{free}$ | $SO_3$ | LOI* | IR** |
|---|---|---|---|---|---|---|---|---|---|
| Dune sand | 94,00 | 0,880 | 0,370 | 2,960 | 0,110 | - | - | 1,500 | - |

**(*Loss on ignition and **Insoluble residue)**

### *2.2 Hydrated lime [Clacium Hydroxide – Ca(OH)₂]*

The setting agent $Ca(OH)_2$ used for activation slag cement is used in the proportions of 0%, 2%, 4%, 6%, 8% and 10% by various methods (substitution and addition by mass of slag cement). The information on the physical properties of the mineral activator (hydrated lime) used is given in Table 3. Its chemical composition is shown in Table 4.

Table 3. Characteristics of the hydrated lime used in this study

| Materials | Absolute density | Apparent density (Kg/l) | Specific surface area (fineness) $cm^2/g$ |
|---|---|---|---|
| Hydrated lime | 2,32 | 0,54 | 8150 |

Table 4. Chemical composition (%, by weight) of hydrated lime used

| Oxides % | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $K_2O$ | $Na_2O$ | $SO_3$ |
|---|---|---|---|---|---|---|---|---|
| Hydrated lime | 0,46 | 0,22 | 0,35 | 68,56 | 0,65 | 0,05 | 0,03 | 0,24 |

The hydrated lime used in this study is a calcium lime: MgO < 5%.

$$\underset{\text{Limestone}}{CaCO_3} \rightarrow \underset{\text{Quicklime}}{CaO} + \underset{\text{Dioxide of carbon}}{CO_2}$$

$$\underset{\text{Calcium oxide}}{CaO} + \underset{\text{Water}}{H_2O} \rightarrow \underset{\text{Calcium hydroxide}}{Ca(OH)_2} + \underset{\text{Heat of hydration (Hydrated lime)}}{Q}$$

### *2.3 Cements*

The Portland cement (CEM II) with mineral addition (slag) was used in this experimental study. Its mineral composition is shown in Table 5. In this experimental study, the setting agent $Ca(OH)_2$ used for activation slag cement is used in the proportions of 0%, 2%, 4%, 6%, 8% and 10% by various methods (substitution and addition by mass of slag cement). Table 6 presents the chemical composition of mineral additions used for the cement studied.

Table 5. Mineral admixture of cement studied

| **Ciments used CEM II** | **Clinker (%)** | **Set Regulator “Gypsum” (%)** | **Admixtures «Slag» (%)** |
|---|---|---|---|
| Hdjar-Soud | 70 | 5 | 25 |

Table 6. Chemical composition of mineral additions (slag and gypsum)

| **Constituents %** | **$SiO_2$ (%)** | **$Al_2O_3$ (%)** | **$Fe_2O_3$ (%)** | **CaO (%)** | **MgO (%)** | **$K_2O$ (%)** | **$Na_2O$ (%)** | **$SO_3$ (%)** | **LOI (%)** | **$H_2O$ (%)** |
|---|---|---|---|---|---|---|---|---|---|---|
| Gypsum | 8,06 | 2,53 | 1,04 | 30,97 | 2,58 | 0,38 | 0,07 | 32,64 | 21,86 | 14,92 |
| Slag | 36,00 | 737 | 4,44 | 42,73 | 4,15 | 0,41 | 0,16 | 1,37 | 1,01 | - |

The chemical composition of the eleven types of cements used in this research have been determined by the testing method "X-ray Fluorescence Spectrometry (XRF)". Tables 7 and 8 give the mixes of the eleven cements activated with calcium hydroxide [$Ca(OH)_2$] by different methods (substitution and addition by mass of slag cement) and the chemical composition of the various cements used in this experimental work.

Table 7. Mix composition of eleven cements activated by the calcium hydroxide

| **Mix of cements** | **Cement “CEM II”, %** | **Lime “$Ca(OH)_2$”, %** |
|---|---|---|
| $CEM_{00}$ | 100 | 0 |
| $CEM_{+2}$ | 100 | 2 |
| $CEM_{+4}$ | 100 | 4 |
| $CEM_{+6}$ | 100 | 6 |
| $CEM_{+8}$ | 100 | 8 |
| $CEM_{+10}$ | 100 | 10 |
| $CEM_{-2}$ | 98 | 2 |
| $CEM_{-4}$ | 96 | 4 |
| $CEM_{-6}$ | 94 | 6 |

| | | |
|---|---|---|
| $CEM_{-8}$ | 92 | 8 |
| $CEM_{-10}$ | 90 | 10 |

Table 8. Chemical composition of eleven cements studied

| Oxides % | $SiO_2$ (%) | $Al_2O_3$ (%) | $Fe_2O_3$ (%) | CaO (%) | MgO (%) | $K_2O$ (%) | $CaO_{free}$ (%) | $SO_3$ (%) | LOI (%) | LSF (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| $CEM_{00}$ | 23,0 | 5,9 | 2,9 | 58,7 | 1,5 | 0,7 | 0,4 | 2,4 | 2,4 | 80,2 |
| $CEM_{+2}$ | 22,3 | 5,6 | 2,8 | 59,2 | 1,6 | 0,7 | 0,8 | 2,3 | 3,0 | 83,7 |
| $CEM_{+4}$ | 21,9 | 5,6 | 2,8 | 59,9 | 1,5 | 0,7 | 2,1 | 2,3 | 3,3 | 86,1 |
| $CEM_{+6}$ | 21,4 | 5,4 | 2,8 | 60,4 | 1,5 | 0,7 | 2,6 | 2,3 | 3,8 | 89,2 |
| $CEM_{+8}$ | 20,7 | 5,1 | 2,7 | 60,8 | 1,5 | 0,6 | 3,2 | 2,2 | 4,1 | 92,5 |
| $CEM_{+10}$ | 20,6 | 5,0 | 2,8 | 61,9 | 1,5 | 0,6 | 3,8 | 2,2 | 4,5 | 94,3 |
| $CEM_{-2}$ | 21,9 | 5,4 | 2,8 | 59,0 | 1,5 | 0,7 | 0,8 | 2,3 | 3,0 | 85,2 |
| $CEM_{-4}$ | 21,6 | 5,3 | 2,7 | 59,2 | 1,5 | 0,7 | 1,8 | 2,3 | 3,4 | 87,2 |
| $CEM_{-6}$ | 21,1 | 5,1 | 2,7 | 60,1 | 1,5 | 0,6 | 2,1 | 2,2 | 4,0 | 90,1 |
| $CEM_{-8}$ | 20,5 | 5,0 | 2,8 | 60,6 | 1,5 | 0,6 | 2,9 | 2,1 | 4,4 | 93,3 |
| $CEM_{-10}$ | 19,9 | 4,9 | 2,6 | 61,3 | 1,5 | 0,6 | 3,3 | 2,1 | 4,6 | 94,5 |

* LOI : Loss in ignition
* LSF : Lime Saturation Factor

The finenesses of the eleven hydraulic cements with minerals admixtures studied was determined by Air Permeability Apparatus.

## 3. Mechanical Tests: Flexural And Compressive Strengths

The mortars samples were subjected to flexural and compressive mechanical tests. Mechanical strength was determined at 7, 28 and 90 days on $4\times4\times16cm^3$ prisms specimens with 50% water-cement ratio and 1:3 cement/Sand (By mass). The moulds with fresh mortar test specimens were cured for 24 h at relative humidity of 95% RH. Three specimens were tested per specimen age.

## 4. Results and Discussion

### *4.1 Influence of the mineral activator (hydrated lime) on the specific surface area (S.S.A) and the specific weight of cement powder*

The Figure 1 presents the effect of mineral activator on the specific weight of cement. From the results obtained (Figure 1), the following conclusions may be drawn:

- a significant difference of the specific weight between the various methods (substitution and addition by mass of slag cement studied).
- a reduction of the specific weight with the increasing of the percentage of the mineral activator (hydrated lime).

The difference observed between the specific weights of activated cements, depends of the content of the mineral activator incorporated in the cement (difference of the density of the mineral activator). The cement activated by the addition method presents the specific weights definitely higher compared to the cement activated by the substitution method, this is mainly due at the variation of the quantity of hydrated lime in each type of cement and with the specific surface area (fineness) of the mineral activator used (very high fineness of hydrated lime).

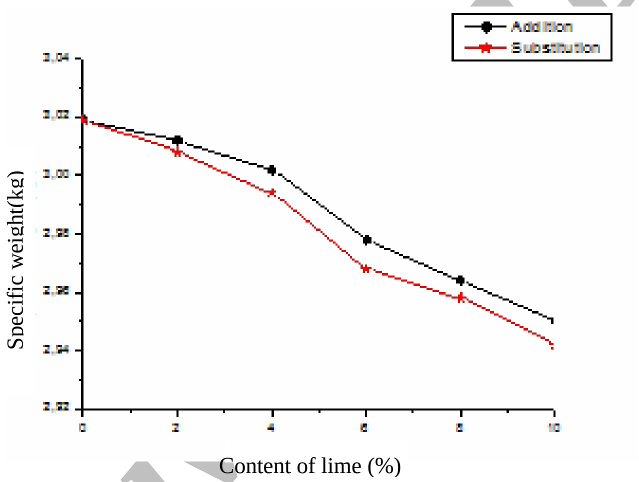
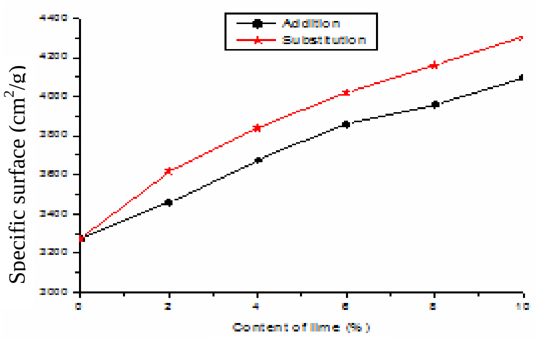



Figure 1. Effect of the mineral activator on the specific weight of cement powder

The Figure 2 shows the effect of the mineral activator on the specific surface area of cement powder. The activation method by hydrated lime of slag cement influences the specific surface area (fineness) largely of cements studied. The increase of the percentage of the mineral activator by various methods (substitution and addition by mass of slag cement studied) increases the specific surface area of cement (variation of the porosity of cement). The difference observed between the specific surfaces area of activated cements, depends of the percentage of the mineral activator incorporated in the cement (high specific surface area of the mineral activator).




Content of lime (%)

Figure 2. Effect of the mineral activator on the specific surface area of cement powder

***4.2 Influence of the mineral activator (hydrated lime) on the cement paste studied***

The Figure 3 presents the effect of the mineral activator on the normal consistency of cement paste. The water demand is measured using the Vicat needle test (standart Vicat test). The influence of the mineral activator on the cement paste is expressed by the changes in normal consistency (water demand ratio).

One notices also that the granulometry of cement (specific surface area) has a significant influence on the normal consistency of cement paste (water demand ratio), this is translated by increase of the total surface of the particles when the cement is ground more finely.

The initial and final set times of cement paste are shown in Figures 4 and 5. When the content of lime increased of cement activated by the mineral activator (hydrated lime), the initial and final setting times of cement paste are decreased (high fineness of cement activated). In general, the set time of cement paste is shortened with the increase of fineness of cements studied (very high fineness of hydrated lime). That is explained by the fact that the pozzolanic reactivity is accelerated in the short-term. The kinetics of hydration of the binder becomes increasingly fast according to the increase of the Blaine fineness (specific surface) of cement.

Indeed, the very fine particles adhere the some to the others and activate the phenomenon of set time of cement paste. Thus the effect of the great Blaine specific surface on the acceleration of the pozzolanic activity reacts with the calcium hydoxide [$Ca(OH)_2$, Portlandite] to form C-S-H gel crystals. The pozzolanic reaction is: [$Ca(OH)_2+SiO_2+H_2O \rightarrow$ C-S-H].

***4.3 Influence of the mineral activator (hydrated lime) on the flexural and compressive strengths of mortar***

The developments of flexural and compressive strengths of the test specimens are shown in Figures 6 and 7. The flexural and compressive strengths increases with curing time for all hardened cement mortars and gives higher values. That is explained by the increase of the fast kinetics of hydration of the mineral $C_3S$ (tricalcium silicate) and $C_2S$ (dicalcium silicate). These latter are the two principal minerals which ensure the development of the resistances to short and medium-term.

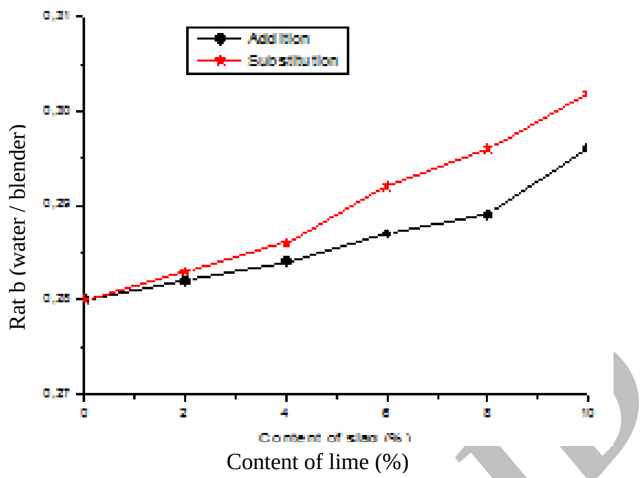



Figure 3. Effect of the mineral activator on the normal consistency of cement paste

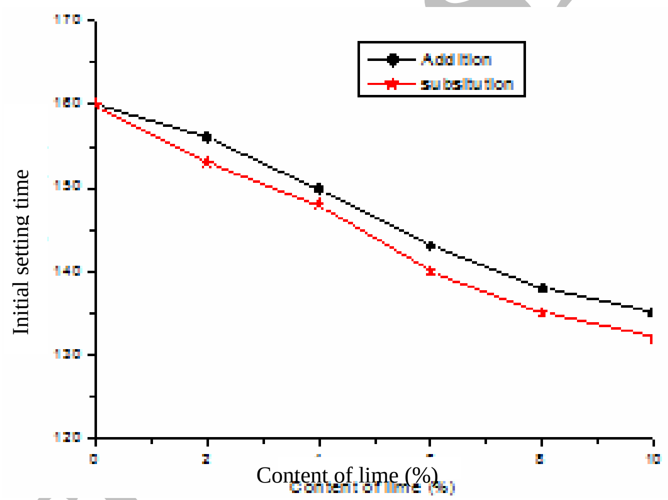
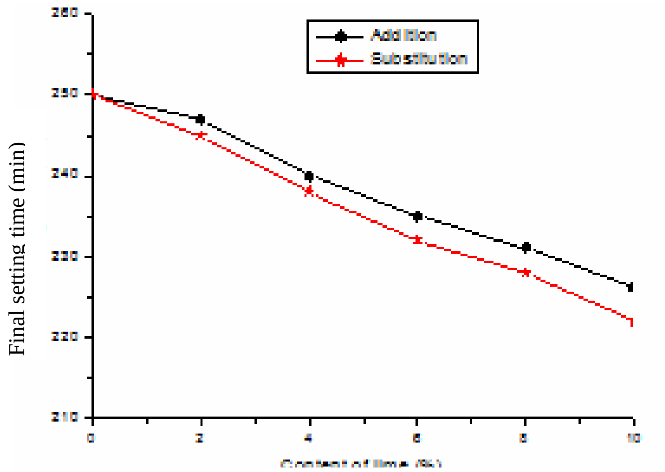



Figure 4. Effect of the mineral activator on the initial setting time of cement paste




Content of lime (%)

Figure 5. Effect of the mineral activator on the final setting time of cement paste

The activation method by the mineral activator of the cement clearly improves the mechanical strengths of the mortar. This confirms the role of the granulometry (mineral activation) in the fast and complete hydration of the cement (pozzolanic activity) by the formation of the $Ca(OH)_2$ released during the hydration of the cement. This pozzolanic reaction gives the second C-S-H supplementary, main responsible for the hardening of the mortar. Therefore the weakness of the strengths to the short-term can be compensated by mechanical activation of cement (increase of the fineness of the cement activated by hydrated lime).

The increase of the mechanical responses as a function of the variation of the fineness (mineral activation) believes a way different of a cement to another cement, this depends of the percentage of the mineral activator (reactivity of the admixture) incorporated in the cement.

Thus, one can conclude that the fineness of cement activated is a significant characteristic: during the hydration of the mixture, more the particles are fine, more the cement surface in contact with water is large and more the hydration is fast and complete (shortening of set times).

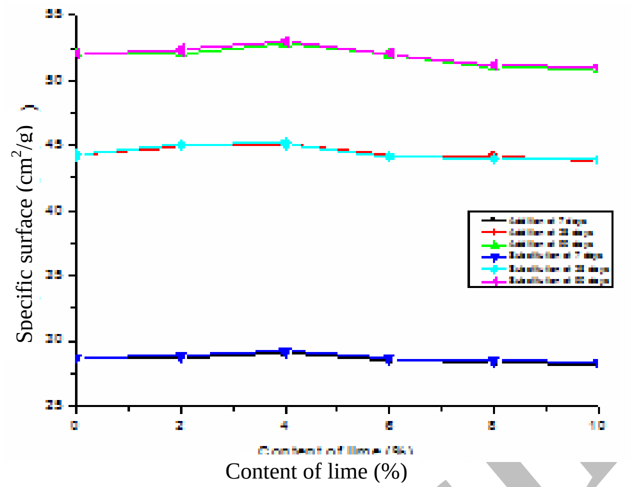



Figure 6. Evolutions of compressive strength of mortars as a function of mineral activator (hydrated lime)

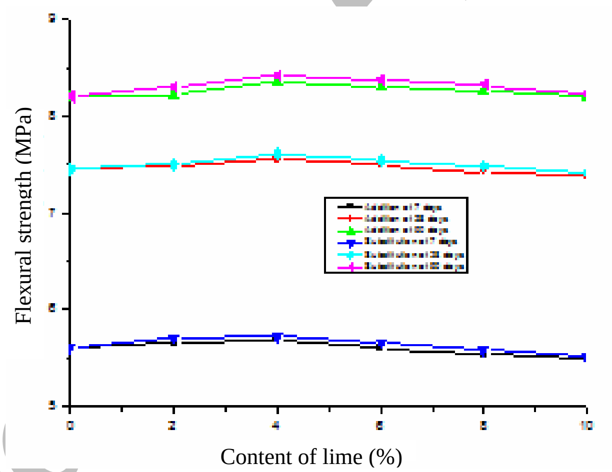



Figure 7. Evolutions of flexural strength of mortars as a function of mineral activator (hydrated lime)

## 5. Conclusion

The results obtained from this research, allow us to draw the following conclusions:

- the activation method by the mineral activator (hydrated lime) of slag cement (latent hydraulicity) influences appreciably on the water demand necessary to have a normal consistency of cement paste.
- the setting times (initial and final) decrease proportionally with the increase of the percentage of lime of cement activated by the mineral activator (hydrated lime),
- the mineral activation (very high fineness of hydrated lime) of slag cement (latent

hydraulicity) presents two essential advantages : high mechanical strength of the mortar as well as a kinetics of hydration reaction accelerated at the initial hardening (short-term).

This is also due to the high fineness of hydrated lime and the percentage of the calcium oxide in cement activated, which accelerate the hydration process, leading to fast setting.

Generally, the activation method by the mineral activator of slag cement (latent hydraulicity) accelerates the hydration process and reduces the setting times of the cement activated.

One can be concluded that, the mineral activator by substitution and addition by mass of slag cement (latent hydraulicity) with hydrated lime up to 4% has a significant effect on the flexural and compressive strengths. However, the mineral activator content of more than 4% adversely affects the mechanical response. The optimum percentage of the mineral activator (hydrated lime) ranges between 2 to 4%. Beyond 6 to 10% of the percentage of the mineral activator, the the mechanical strength of the slag cement decreases.

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