# EFFECT OF ALKALI-ACTIVATION IN THE MICROSTRUCTURE, SETTING-TIME AND COMPRESSIVE STRENGTH OF PORTLAND CEMENT PASTES AND MORTARS

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### ABSTRACT

This work presents a study about alkali-activation of Portland cement pastes and mortars by  $Na_2O$ ,  $K_2O$ ,  $Na_2SiO_3$ and  $K_2CO_3$ , in proportions of 0, 3, 5, 10 and 15%, in weight. The aim was correlate the microstructures formed with the properties developed in the fresh and hardened stages. The results show that the alkalis changes the chemical equilibrium of all hydrates formed, and this promotes lower compressive strength in final ages. Quickly setting times and early-high compressive strengths have been obtained with alkalis, but as  $Na_2SiO3$  and  $K_2CO_3$  were more efficient.

### 1. INTRODUCTION

The alkali-activation of granulated blast furnace slag is developed with successful since half of  $20^{\text{th}}$  century, in several countries of the world [1]. The potentiality of the use of alkalis opens new opportunities to obtain special cements, with different properties from those presented by ordinary Portland cement [2]. When ionic species, in solution, are incorporate in the mixture water, its increase the speed of dissolution of Portland cements anhydrous compounds, working as catalysts [3]. The mobility of OH and Ca<sup>2+</sup> ions is changed, and those combinations occurs more quickly. The use of metallic alkalis ions, as Na+ and K+, for acceleration of hydration of the Portland cement and its microstructural effects in post-hydration is the aim of this work.

#### 2. MATERIALS AND METHODS

The Portland cement used was the CPIIE-32 (NBR 11580) supply by Lafarge S/A. The activators  $Na_2O$  and  $K_2O$  were obtained by its respective hydroxides, both P.A. (Vetec S/A). The  $Na_2SiO_3$  was a commercial grade with  $SiO_2/Na_2O$  ratio of 2.74 and the  $K_2CO_3$  was P.A. (Reagen S/A). All activators were used in proportions of 3, 5, 10 and 15% wt by wt in relationship the Portland cement. The setting times were determined by Vicat needle (NBR 7215). Four Normal mortar specimens were molded for compressive strength

tests (NBR 7215). Samples of hydrate pastes were selected to microstructural analysis by low vacuum scanning microscopy (SEM - Jeol 5800 LV). The hydration process was interrupted by acetone immersion before gold sputtering. The compressive specimens were cured by water immersion ( $25 \pm 2$  °C) until the date of tests.

#### 3. RESULTS AND DISCUSSION

The results showed that the alkali ions very modify the kinetics of hydration of Portland cement. As was expected, the setting times of all samples were decreased. The Figure 1 shows the setting time accelerating effect of the alkalis. The early compressive strengths are increased more than 300% at 1 day of age. However, at ages later (28 and 90 days) the compressive strengths were inferior to the values developed by the reference specimens (Figure 2).



Figure 1 - Variation of setting times with activators content.

The Figure 3 shows details of microstructure developed at 1 day of age with 5% of Na<sub>2</sub>O. The microstructure reveals the presence of lamellae crystals with rounded edges, rich in CaO (65,8%), Fe<sub>2</sub>O<sub>3</sub> (15,4%) and Na<sub>2</sub>O (4,3%), and also shows low contents of SiO<sub>2</sub> (8,3%) and Al<sub>2</sub>O<sub>3</sub> (3,8%), determined by semi-quantitative analysis (EDS).

These crystals, of 5  $\mu$ m of dimension, growing between small agglomerates formed by particles of colloidal

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dimensions (< 1  $\mu m)$ . The microstructures developed by alkali-activated paste with  $K_2CO_3$  is show in Figure 4.



Figure 2 - Compressive strengths until 90 days of age to 5% of different activators.



Figure 3 - SEM micrograph of paste with 5% Na<sub>2</sub>O, at 24 hours of age (5000X).



Figure 4 - SEM micrograph of paste with 5% K<sub>2</sub>CO<sub>3</sub>, at 24 hours of age (5000X).

The pastes activated with  $K_2O$  presented high potassium contents in all hydrate phases, competing with the calcium, in ratios up to 1:1. In the C-S-H phases, the (CaO+K<sub>2</sub>O)/SiO<sub>2</sub> average ratio, oscillated around 2.5.

The lower strength observed with the alkali ions add, is associated to the precipitation and crystallization of alkalis into the matrix, breaking its homogeneity and generating osmotic tensions that contributes by multiple microcracking of matrix. The Figure 5 shows the precipitated particle of KOH in the paste with 5%  $K_2O$  at 28 days of age.

The paste activated by 5%  $Na_2SiO_3$  reveals at 28 days of age a porous microstructure with the presence of acicular AFM particles, identified as  $CAH_{10}$  [3]. However, this activator promoted the best results of later compressive strengths (Figure 6).



Figure 5 - SEM micrograph of paste with 5% K<sub>2</sub>O at 28 days of age (5000X).



Figure 6 - SEM micrograph of paste with 5% Na<sub>2</sub>SiO<sub>3</sub>, at 28 days of age (5000X).

# 4. CONCLUSIONS

High contents of alkali activator (10-15%) are deleterious for all types of activator, principally Na<sub>2</sub>O and K<sub>2</sub>O. When alkali ions were add to water mixture, the setting times are shorter and the later strengths are lower. However, 5% of Na<sub>2</sub>SiO<sub>3</sub> addition gave high early compressive strengths (400% more than developed by reference specimens). In addition, the final compressive strength developed by mortar specimens (90 days) with Na<sub>2</sub>SiO<sub>3</sub> is so 20% smaller than reference specimens. The alkali activation with K<sub>2</sub>CO<sub>3</sub> showed similar results to Na<sub>2</sub>SiO<sub>3</sub>. Therefore, the use of alkali in the combined forms as Na<sub>2</sub>SiO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub> shows be viable to activate Portland cements pastes and mortars.

# 5. ACKNOWLEDGEMENTS

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# 6. REFERENCES

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