Beneficial Effect of Incorporation of Slag on the Hydration Heat, Mechanical Properties and Durability of Cement Containing Limestone Powder

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BENEFICIAL EFFECT OF INCORPORATION OF SLAG ON THE HYDRATION HEAT, MECHANICAL PROPERTIES AND DURABILITY OF CEMENT CONTAINING LIMESTONE POWDER

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ABSTRACT

This paper presents the experimental results of a wide research program, tending to determine the hydration mechanism, mechanical properties and durability performance in ternary cement containing limestone powder and blast furnace slag. The limestone powder increase the hydration at early ages inducing a high strength at early age, but it can reduce the later strength due to the dilution effect. On the other hands, Granulate Blast Furnace Slag (GBFS) contributes to increase the compressive strength at later ages. Hence, at medium blended cement (OPC-LP-GBFS) with better performance could be produced. Results show at an early age the limestone powder, increase the hydration degree and the compressive strength. At later age the Granulate Blast Furnace Slag is very effective in producing ternary blended cements with similar on higher compressive strength than the cement (CEM II/A 42.5) at 28 and 90 days. For durability, the incorporation of the blast furnace slag into the limestone cement improves remarkably resistance effect to attack by acids and sulfates and it has been found that the durability of the cements never depends on the mechanical strength.

Keywords - Acid attack, Compressive strength, Hydration heat, Mineral addition, Mortar, Sulfate attack, Acid attack.

I. INTRODUCTION

The laboratories research in materials are working on the development of new composite cementitious, in order to improve the mechanical properties and durability especially in aggressive environments. The resistance of concrete to sulfate attack is one of the most important factors to ensure its sustainability. Sulfate reacts with the aluminates by forming complexes sulfoaluminates of calcium by decomposition of the chloroaluminates of calcium.

The sources of sulfates products are internal or external to the concrete. In the first case, the sulfates come from; the soils, seleniteuses groundwater and the water of sea. The destruction mechanisms of sulfates are based on the concentration and source ions sulfates in the external water solution or in cement paste. When the concrete will crack, the permeability increases and aggressive water penetrates easily, which accelerates the pace of destruction.The addition of limestone powder to clinker completes the fraction in the granulometric curve of cement without an increment on water demand, improves the cement packing and blocks the capillary pores. Large number of studies [1.2.3] have show that granulated blast furnace slag (GBFS) have been widely used as a cement replacement in Portland cement because of their advantageous for improving mechanical and durability concrete properties and for bringing environmental and economic benefits.

It should be possible by the systematic adjustment of the proportions, to produce ternary blended cement (OPC-LP-GBFS) which utilizes the desirable characteristics of one addition while compensate for the undesirable characteristics of the other. The limestone powder contributes to the increase strength at early and the granulated blast furnace increase the long term. The presence of blast furnace slag in the cement gives lower hydration heat compared to that of ordinary cement; this is due to its latent hydraulic reaction [4]. On the other hand, the best combination of these mineral additions, can lead to an enhanced durability performance.

On attacks to sulfates, Gonzalez and Irassar [5] reported that the addition of 10% of limestone reduced the expansion by 30% after 6 months exposure to 5% Na_2SO_4. The reduction in expansion for limestone specimens is probably because of the formation of monocarboaluminate in the presence of limestone and hence the suppression or delay of the conversion of ettringite to monosulfoaluminate.

Zivika and Bajz [6] reported that some authors have claimed that the use of pozzolana had only a weak positive effect on the increase of the acidic resistance.

In Algeria most of the cement is being blended with additions such as limestone and granulated blast furnace. The cement plants usually add about 20% of GBFS and 10 % of limestone filler as cement replacement by weight. Effete of utilizing both LF and GBFS on concrete properties is not well cemented in the literature.

Granulated blast-furnace slag (GBFS), either as a constituent of cement or as a mineral admixture, is widely used to make not only traditional concrete but also high-performance concrete, which has several advantages in terms of workability, long-term strength, and durability [3.7].The hydration heat of blast furnace slag is initially lower than that of Portland cement. Thereafter, Portland cement containing blast-furnace slag typically shows a reduction of strength at early ages and similar or greater strength at later ages [8]. Rao et al. studied the Abrasion resistance and mechanical properties of roller compacted concrete with GGBFS. They also found that abrasion resistance of roller compacted concrete with ground granulated blast furnace slag (GGBFS) as mineral admixture was strongly influenced by its compressive strength irrespective of GGBFS content [9].

Generally speaking, replacement of slag for cement was found to lead to reduced water absorption; this reduced absorption was observed to rise with increasing cementitious materials from 12 to 15%. Overall, water absorption in all the mix designs investigated was found to be less than 4% [10].

The objective of this paper is to present the optimization of the compressive strength and the durability performance in ternary cement containing limestone filler and granulated blast furnace slag. The degree hydration of mortar containing ternary blended cement (OPC-LF-GBFS) is also presented in this study was measured, up to 5 days and the mechanism hydration is discussed.

II. EXPERIMENTAL

A. Material properties, mortar mixture proportion

The cement used in this study is blended cement (CEM II/A 42.5) containing 10% of limestone powder. The granulated blast furnace slag was provided by El. Hadjar steel factory (situated in the east of Algeria). The Blaine specific surface for cement and the addition was measured with Blaine method according to NF EN 196-6 [11] and is given in Table 1. The chemical compositions and physical properties of the materials used are summarized in Table1. The mineralogical composition of blended cement (CEM II/A 42.5) is calculated by Bogues formula, was C_3S = 41.8%, C_2S = 33.3%, C_3A = 5.1% and C_4AF = 10.7%.

TABLE 1

The chemical compositions and physical properties of the materials used.

	CEMII/A 42.5	**Blast-furnace slag**
SiO_2	22.6	42.20
Al_2O_3	4.20	6.85
Fe_2O_3	3.56	1.90
CaO	62.18	42.20
MgO	0.63	4.72
SO_3	2.2	1.54
Na2O	/	0.12
K2O	0.42	0.43
Loss of ignition	1.84	0.80
Specific density Blaine Finesses cm^2/g	3.1 3200	2.9 3400
Glass content, %	/	/

The basicity coefficient of GGBS and LP was calculated based on the chemical composition and is determined by equation (1) and this values reported in Table 2.

$$Ib = \frac{(1-p)(CaO+MgO)_{cement} + p(CaO+MgO)_{addition}}{(1-P)(SiO_4+Al_2O_3)_{cement} + p(SiO_4+Al_2O_3)_{addition}} \qquad (1)$$

TABLE 2

values of basicity coefficient for different type of substitution

Cement type	**Basicity coefficient**
10% LP	2,34
10% LP +10% GBFS	2,11
10% LP +20% GBFS	1,92
10% LP +30% GBFS	1,75
10% LP +40% GBFS	1,59

Eleven blended cements were formulated by the addition in the cement (CEM II/A 42.5), Granulate blast furnace slag, from 0% to 40%. All replacements were made by mass. Table 3 shows the combination of blended cements studied. Sand/cement ratio is equal to 3 and water/cement ratio is 0.50. For all mixtures prepared were mixed according to the ASTM C305-06 standard [12]

B. *Testing*

The hydration heat is measured by a semiadiabatic calorimeter according to the NF EN 196-9 standard [13]. The method consists of quantifying the heat generated during cement hydration using a thermally insulated bottle. The test is prepared at laboratory temperature of 20 °C. The measurements are made over 120 h. The total hydration heat is calculated by the following expression (2):

$$q(t) = \frac{C}{m_c}\Delta\theta + \frac{1}{m_c}\int_0^t \alpha\Delta\theta dt \quad (2)$$

Where:

C is the total thermal capacity of the mortar filled calorimeter (J/°C), mc is the cement or powder mass (g), Δh is the mortar temperature rise (°C), and a is the total calorimeter thermal loss coefficient (J/h °C).

The strength of mortars was determined in accordance with the European standard EN196-1[14]. The mortar was placed in 4×4×16 cm^3 -prismatic steel moulds. After casting, specimens were left covered with a plastic sheet. After removal from the moulds, at 24 h of age, mortar specimens were immersed in water saturated with lime at 20°C until the age of testing were conducted at 2, 7, 28 and 90 days of age. The flexural strength of mortars was evaluated by three-point bending tests carried out on 4×4×16 cm^3 prismatic specimens, with a loading speed of 50 N / s. The two parts of each 4×4×16 cm^3 mortar sample first subjected to a three-point bending test are reused for a compression test.

The durability was conducted only on control cement and the cement containing 10% of limestone powder, 10%, 20%, 30% and 40% of granulate blast furnace slag. For the sulfate attack tests, the mortars specimens were immersed in 5% sodium sulfate (Na_2SO_4) or 5% magnesium sulfate ($MgSO_4$) at laboratory temperature 20°C .The sulfate attack was evaluated through the measurement of the expansion on the prismatic specimens measuring 4×4×16 cm^3. The relative acid attack was determined in accordance with ASTM C-267[15]. The mortars specimens measuring 4×4×4cm^3, were cured in water at 20°C for 28 days before subjected to acid attack. Three specimen of each mortar mix were immersed in 3% sulfate acid (H_2SO_4) or 1%hydrochloric acid (HCL). The solution was renewed every 15 days and the weight loss of the specimens measured. The attacked portions of the mortar specimens were cleaned with deionizer water and then the acid attack was evaluated through measurement of the weight loss of the specimens determined as follows:

$$Weightloss(\%) = \frac{M_2 - M_1}{M_1} \times 100 \qquad (3)$$

Where M1 is the weight of the specimen before immersion and M2 is the weight of the cleaned specimen after immersion.

IV. RESULTS AND DISCUSSION

A. Material properties, mortar mixture proportion

Here comes the most crucial step for your research publication. Ensure the drafted journal is critically reviewed by your peers or any subject matter experts. Always try to get maximum review comments even if you are well confident about your paper. The hydration rate of cement based composite mainly depends on three parameters: the cement content, the activity of admixture, and the effect of admixture on the hydration of cement. The results of the hydration heat of mortars are illustrates in Fig.1. The cement containing 10% of limestone powder is characterized by a rapid hydration, which reaches 80% of its final value at the first day and accomplishes its maximum value 98% at the third days. The fine particles of limestone powder can act as heterogeneous nucleation site on which the product of C–S–H gel could precipitate, so limestone powder has a promoting effect on the hydration of cement. Barbara et al. [16] found that the exothermic peak in the end of the acceleration period appeared 1 h head of time and the cumulative heat after 72 h increased by replacing 4 % of cement with limestone powder. This was because the reduction of cement content was insignificant, but the promoting effect of limestone powder was significant. The addition of limestone powder in the cement can increase the rate of hydration, which induces a decrease in the capillary pores [17].

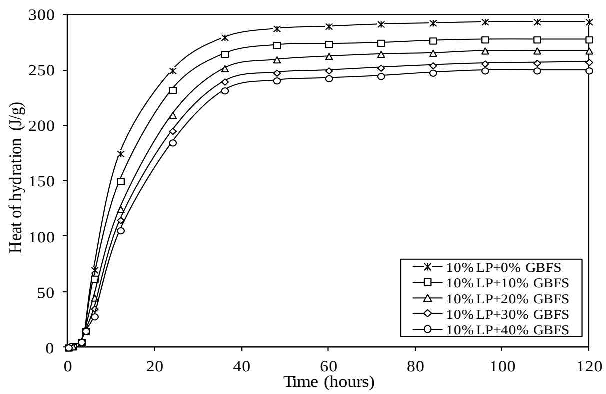

Fig 1 Hydration Heat for Ternary Blended Cement Containing Limestone Powder and Blast furnace Slag at 120 hours

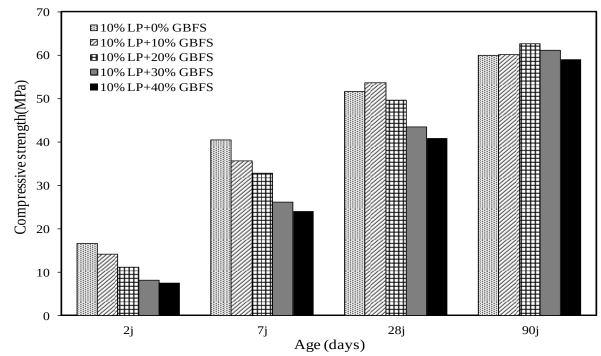

Fig 2 Compressive Strength for Ternary Blended Cement Containing Limestone Powder and Blast furnace Slag at Different Ages

The hydration heat of ternary binders containing, the blast furnace slag and limestone powder are shown in Fig.1. It is clear that the hydration heat of cement blended decreases with increases the percentage of blast furnace slag and is higher than that control cement (90%OPC+10%LP+0%GBFS). For example, the substitution of 10, 20, 30 and 40% of slag in the cement containing 10 % of limestone powder the hydration heat decreases of the order 6, 10, 14 and 17% respectively; this is due to its latent hydraulic of blast furnace slag that reacts at later age [4].

Compared to composite binder containing limestone, lower amount of hydration heat is released for composite binder containing the same amount of slag due to the lower activity of slag. Also, is higher compared by others ternary cements. One can explain these results by the character hydraulicity of blast furnace slag that is manifested belatedly. The exothermic peak in the end of the acceleration period of the binder containing LP is higher than that of the ternary cement containing LP and GBFS, and the exothermic peak of the binder containing limestone powder appears earlier. It is obvious that the exothermic rate of the binder containing limestone powder is much faster than that of the binder containing steel slag in the acceleration period. In the deceleration period, their exothermic rates are close to each other [18].

B. *Development of the compressive and flexural strength*

The results reported in Fig.2 and Fig.3 presents the evolution of compression strength and flexural strength with age of mortars with ternary binders as a function of mineral additions and their substitution rates. It was noticed from these figures the typical development of compression strength for mortars containing 10 % of limestone powder (0%OPC+10%LP) and ternary cement containing 10% of limestone powder and 20% of Granulate Blast Furnace Slag (0%OPC+10%LP+20%GBFS). For filler blended cements compressive strength à generally higher than author’s types of cement at early ages, comparable strength at 7 days and lower strength at later ages.

Fig.2 illustrates the curves of compressive strength showing the interaction effect of the LP and GBFS for the domain studies in the ternary system. At 2, 7 and 28 days, the stationary point corresponding to the maximum compressive strength is obtained by the replacement of 10% of LP and a very little content of GBFS.

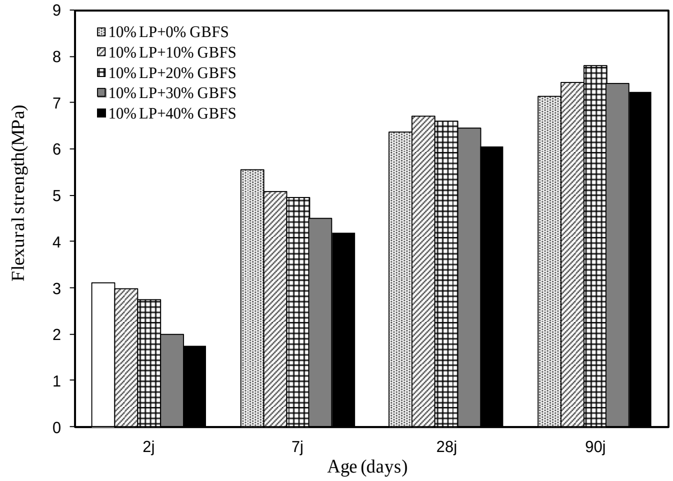

Fig 3 Flexural Strength for Ternary Blended Cement Containing Limestone Powder and Blast furnace Slag at Different Ages

While, the zone of maximum strength is located around to 10% of LP, replacement is low level of GBFS replacement (GBFS 10%). However, the values of compressive strength changes significantly at 90 days, where the maximum compressive strength is obtained with the larger proportion of GBFS (20%) and around 10% of LP. At this point the strength was 5.6 % higher than the corresponding witness cement (10%LP+0%GBFS). In summary, the point of maximum strength is around 10% of LP and low GBFS replacement level at the early ages (2,7 and 28 days), after 28 days, this point moves toward the high level of GBFS replacement and low LF content. These results were similar to the results of others researches [3.19.20]

The relationship between the compressive strength and the flexural strength is given in Fig.4 the correlation between the flexural strength and the compressive strength results were calculated for the several researches of the test results and hence the relation obtained is:

$$f_f = 0{,}5176(f_c)^{0.6501} \qquad (4)$$

It can be observed in Fig.4 the results, where it gives correlation coefficients very close of unity; it is of the order of 0.99.

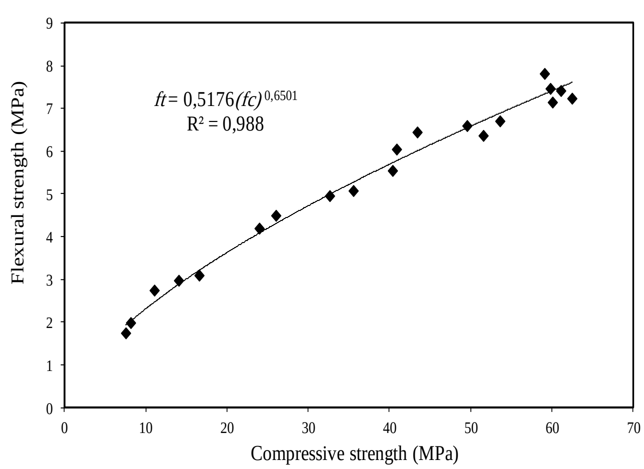

Fig 4 Correlation Between Flexural Strength and Compressive Strength.

C. Durability tests

1) Sulfate attack: The results obtained indicate that specimen expansion increases steadily as a function of immersion time. This results in the appearance of expansive mineralogical phases in the hardened material (gypsum and secondary ettringite) which can swell and crack the cement paste. The quality of gypsum formed in the reaction between sulfates and portlandite ($Ca(OH)_2$), which is responsible for the formation of secondary ettringite, be further, the pozzolanic reaction produces a secondary CHS gel that also since it is deposited in the pores and enhances the paste-aggregate interface[24.25.26]. As soon as the needle crystals have no room for growth in the pores, crystallization pressure occurs, causing the expansion and ultimately the bursting of the concrete surface

Fig.5.a shows the expansion values at 6 months of exposure to $MgSO_4$ Solution are 1.26%, 0.86%, 0.76, 0.71% and 1.1%, for mixes with 0%, 10% GBFS, 20% GBFS, 30% GBFS, 40% GBFS and 10%LF. The addition of 20% to 30% of GBFS produces an important reduction of expansion of 65% to 77% respectively than the corresponding expansion of the (CEM II/A 42.5) mortar and the expansion of mortars immersed in the solution ($N_{a2}SO_4$) reduced to 45 and 90% respectively (see Fig.5.b). These results were similar to the results obtained by Allahvedi et al [21] where they found that alkali-activated slag cement exhibits a higher sulfate resistance compared to Portland cement.

The increase in the percentage of substitution of slag in the cement decreased the expansion because of its basicity index which is weak. These results are in conformity with those reported by other investigators [22.23]

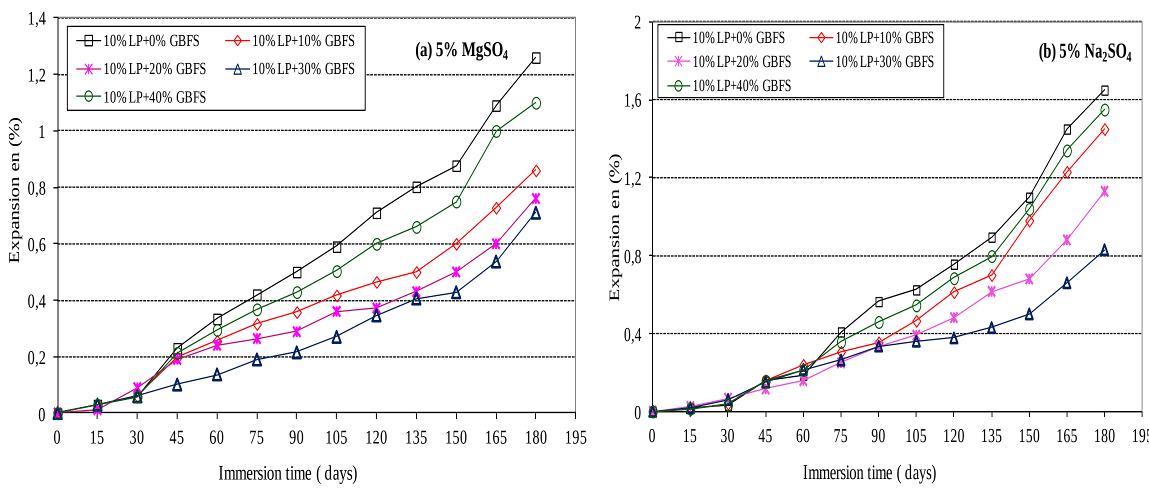

Fig 5 Expansion of mortar exposed to (a) 5%MgSO4 and (b) Na2 SO4

2) Acid attack: As shown in Fig. 6 the test results of weight change versus time for mortar specimen exposed to 3% H_2SO_4 and 1% HCL solutions for 6 months. After 180 days exposure to 1% HCL solution, the total loss in weight of mortar containing (60%OPC+10% LP+30% GBFS) and mortar containing (50%OPC+10% LP+40%GBFS), the loss weights are lower than the corresponding mortar containing (90%OPC+10%LP+0%GBFS) by 26% and 14 % respectively as shown in the Fig.6.a.On the other hand, after 180days exposure to 3% H_2SO_4 solution, the total loss in weight of mortar containing (60%OPC+10%LF+30%GBFS) and mortar containing (50%OPC+10%LF+40% GBFS), the loss in weight is lower than the corresponding (CEM II/A 42.5) mortar by 25% and 28% respectively as shown in the Fig.6.b. From these results, it is noted the beneficial effect of the incorporation of slag in the cement containing the limestone powder, which minimizes the penetration of aggressive agents. So we can conclude that the two combinations (60% OPC+ 10% LF+ 30% GBFS) and (50% OPC+ 10% LF+ 40% GBFS) have stood the acid attacks. The positive influence of the percentage of slag on the chemical resistance is translated on the one hand, by the Pozzolanic reaction which sets the portlandite released during cement hydration to form additional CSH gels of 2nd generation and therefore the compactness of carbohydrates increased, resulting in a reduction of porosity which prevents the penetration of solutions of acids in the cementitious matrix. These results are in conformity with those reported by [27] or it was found the incorporation of 40% of slag reduced porosity, lower water absorption, and concrete permeability. It has been found that the resistance the cements to attack by acid never depends on the mechanical strength, that is to say a cement of good mechanical strength does not always give a cement of good resistance to acid attack as show in the Table 4.

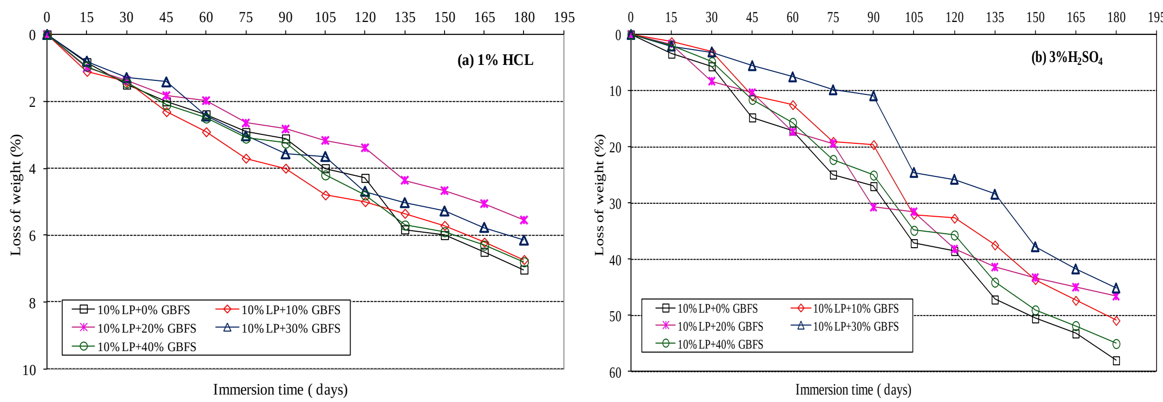

Fig 6 weight loss of mortar due to the acid attack (a) 1% Hcl and (b) 3 % H2SO4

Bakharev et al [28] reported that some authors have claimed that the use of blast furnace slag had only positive effect on the increase of the acidic resistance. Therefore, it can be concluded from these results that granulated blast furnace slag can be used to improve sulfuric acid (H_2SO_4) and hydrochloric acid (HCL) resistance.

IV. CONCLUSION

This study was conducted to assess the effect of the interaction between limestone filler and granulated blast furnace slag for Algerian addition on the properties of mortar. The following conclusion can be drawn:

- The hydration heat of cement blended decreases with increases the percentage of blast furnace slag in the cement containing limestone powder.
- For the strength properties, the combination of limestone filler and blast furnace slag is complementary: the point of maximum strength is around 10 % of limestone powder and low blast furnace slag replacement level at the early ages after days, this point toward the high level of BFS replacement.
- Lower expansion as observed in cement control (CEM II/A 42.5). On the other hand blast furnace slag improved the resistance to sulfate attack or the addition of 20% to 30% of GBFS produces an important reduction.
- It can be concluded from these results that granulated blast furnace slag can be used to improve sulfuric acid (H_2SO_4) and hydrochloric acid (HCL) resistance.
- The use of limestone powder in the hydrochloric acid environment seems to be not recommended.
- The incorporation of the blast furnace slag as addition in cement containing limestone powder is very beneficial to the resistance in sulphuric and hydrochloric acid mediums.
- The durability performance of the cements never depends on the mechanical strength.

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