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Strength optimization of "tailor-made cement" with limestone filler and blast furnace slag

M.F. Carrasco*, G. Menéndez, V. Bonavetti, E.F. Irassar

Departamento de Ingeniería Civil, Facultad de Ingeniería, Universidad Nacional del Centro de la Provincia de Buenos Aires, B7400JWI, Olavarría, Argentina

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Abstract

The use of cements made with portland clinker and two or three additions has grown because they present several advantages over binary cements. Production of composite cements has produced a necessary shift in the manufacture process used in the cement industry. Now, it is known that the separate grinding and mixing technology is more convenient in order to produce these cements, called *market-oriented* or *tailor-made cements*. However, their optimum formulations require the help of methods of experimental design to obtain an appropriate performance for a given property with the least experimental effort.

In this study, the interaction between limestone filler (LF) and blast-furnace slag (BFS) is analyzed in mortars in which portland cement (PC) was replaced by up to 22% LF and BFS. For this proposition, a two-level factorial design was used permitting to draw the isoresponse curves. Results show that compressive and flexural strength evaluated at 2, 7, 14, 28, 90 and 360 days are affected in different ways by the presence of mineral additions.

Keywords: Hydration; Compressive strength; Blended cement; Filler; Granulated blast-furnace slag

1. Introduction

Blended cement is an old component of concrete mixtures. Pozzolanic cement was standardized in Italy in 1929, blast-furnace slag cements were produced in Germany, France, Luxembourg and Belgium for more than a century and cements containing fly ash appeared in France in 1950. However, the importance of mineral additions has notoriously rose in the last decades due to cement industry requirements, as well as the need of longer service life for concrete structures.

The use of mineral additions in cement production implies a reduction in consumption of fossil fuel, mineral natural resources and the gas emissions that contribute to the green-house effect. Consequently, partial replacement of portland clinker is a feasible solution from an economic, ecological and technical point of view. Since the 1970s, many efforts have been done to find appropriate mineral additions from natural resources (pozzolans, limestone filler), heat-activated additions (clay, metakaolin) or industrial by-products (fly ash, blast-furnace slag, silica fume, rice husk ash) [1]. Around the world, several combinations of mineral additions have been used to formulate blended cements depending on the available resources that can be found in each country.

During the 1990s, the use of cements made with portland clinker and two additions, called ternary or composite cements, has grown because they present some advantages over the binary cements [2]. Nowadays, composite cements containing combinations of fly ash and silica fume or slag and silica fume are commonly used and many studies have been carried out on this topic [3,4]. Ternary cements could contribute to achieving the needed balance between the

* Corresponding author. Tel: +54 2284 45 10 55; fax: +54 2284 45 06 28.

E-mail address: lem@fio.unicen.edu.ar (M.F. Carrasco).

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industry's quest for high-performance products and the restrictive environmental regulations. In the ternary cement, the synergistic effects could allow individual component ingredients in such blends to compensate for their mutual shortcomings. They present an opportunity to produce environmentally friendly concrete with tailor-made properties for the requirements of market, without expansive investment cost [5].

In Europe, the EN 197-1 standard identifies two types of portland composite cements according to the replacement level of active mineral additions (slag, silica fume, natural pozzolan, fly ash): type II/A-M containing 6–20% and type II/B-M containing 21–35%. These cements, as other types defined by this standard, can contain a proportion up to 5% of minor additional constituents and they are classified for different strength classes according to the strength gain until 28 days. A similar trend is observed in Latin American countries. Brazilian standard (EB-2138/91) defines the types CPII-E (BFS<34%+LF<10%) and CPII-Z (Pozzolanic material<14%+LF<10%) cements. Composite cements also were standardized in Argentina (IRAM 50000-00) and Mexico (NMX C-414-0/99) admitting the incorporation of two or more mineral additions with a maximum content of 35%. In USA, ASTM C 1157 standard has introduced performance-based hydraulic cements that do not limit the type and the amount of mineral additions that can be blended with portland cement.

Composite cements can be formulated allowing the compensation of mineral additions shortcomings by the synergistic effect produced in ternary blends. For these cements, the basis is usually clinker plus BFS and a third addition such as pozzolan, metakaolin, fly ash, silica fume or limestone filler. They can be produced by intergrinding process or by the grinding and mixing technology. The intergrinding process is easier, technologically simple and the homogenization is made in the grinder device. However, the particle size distribution (PSD) of composite cements obtained by this process depends primarily on the grindability of each component. The second technology consists in grinding and storing separately the components (clinker+gypsum, slag, limestone) and finally mixing them in previously determined proportions to obtain a composite cement according to user requirements. This process has several advantages such as control of particle size distribution of each component, choice the PSD parameter according the role of each component, choice the appropriate technology or process for milling hard or soft component and it is an economic way to produce small amounts of different type of cements according to the market requirement [6], commonly called *tailor-made cement* [5] or *market-oriented* [6].

In general, fillers are incorporated in order to complete the granulometric distribution of cement decreasing the water demand, to enhance its granular packing factor and to block up capillary pores. Moreover, filler particles accelerate the hydration of silicate and alumina phases of clinker grains acting as nucleation centers for CH crystals and, as a consequence of this chemical and physical interaction, increasing the early strength of cement [7,8].

Pozzolanic and cementitious materials, when mixed with portland clinker and water, produce C–S–H similar to that generated from the hydration of calcium silicate of clinker [9]. This reaction is slow compared with that of the portland cement, leading to a lower strength at early ages and similar or higher values at later ages [10]. Also, investigations and practical experiences show that fly ash, natural pozzolan and BFS could increase the water demand [6].

For the optimum formulation of these cements, methods of experimental design can be used to determine the influence of mineral addition proportions on the development of strength and other properties of cement analyzing its multifactorial dependencies. These methods highlight the significance of the effect of experimental variables and their interactions and they present a predictive capability for the response of other experimental points located within the experimental domain. They were successfully applied to find out the optimum proportion of mixtures containing mineral additions for several properties of paste, mortar or concrete [11–13].

According to the literature review, the combination of LF and BFS in composite cement can help to formulate a cement with an adequate development of strength, because LF contributes to the early strength and BFS increases the long-term strength. On the other hand, experimental design can lead to find the best combination of these mineral additions to obtain an appropriate response for a given property with the least experimental effort.

The objective of this paper is to provide information for the optimization of the compressive and flexural strength in composite cements containing limestone filler and blast-furnace slag.

2. Experimental

2.1. Materials

A portland cement (PC), limestone filler (LF) and blast-furnace slag (BFS) were used in this investigation. The chemical composition obtained by X-ray fluorescence spectrometry, and the physical characteristics of these materials are reported in Table 1. The mineralogical composition (Bogue) of portland cement was C_3S=59.2%, C_2S=17.5%, C_3A=4.5% and C_4AF=9.6%. According to XRD analysis, LF contains 87% of $CaCO_3$ in calcite form and the main crystalline impurity is quartz. As results of chemical analysis, BFS has a chemical modulus ((C+M+A)/S) of 1.73 and its XRD pattern showed the absence of crystalline compounds and a hump centered on the main peak of melilite at $2\theta=30.0^\circ$ (d=3.00), characteristic of well

Table 1
Chemical composition and physical characteristics of materials

	Portland cement	Blast-furnace slag	Limestone filler
Chemical composition (%)			
SiO_2	21.66	35.50	8.70
Al_2O_3	3.66	12.52	0.81
Fe_2O_3	3.07	0.54	0.49
CaO	64.30	38.96	49.08
MgO	0.67	9.88	0.50
SO_3	3.16	0.54	0.19
K_2O	1.13	0.43	0.25
Na_2O	0.15	0.07	0.18
Loss by ignition	1.29	0.05	39.20
Physical characteristics			
Specific gravity	3.11	2.88	2.68
Fineness (Blaine) (m^2/kg)	297	438	522
Retained on sieve (%)			
75 μm (#200)	1.1	0.0	6.3
45 μm (#325)	12.8	0.3	24.4
Position parameter, x' (μm)[a]	26.93	17.26	28.56
Homogeneity parameter, n[b]	0.92	1.05	0.57

[a] Characteristic diameter of particle size distribution obtained at a cumulative mass of 63.2%.

[b] Slope of particle size distribution curve representing the wide of distribution.

quenched slag [14]. The activity index of BFS was 78% at 7 days and 111% at 28 days, indicating a very active slag according to EN-196-1 standard. The specific surface Blaine and PSD parameters, obtained by a laser granulometer, are also reported in Table 1.

Nine composite cements (binary and ternary) were formulated replacing portland cement (PC) by up to 22% of LF and/or BFS (by mass) selected according to the experimental plan described bellow. Additionally, mortar made with PC was evaluated and taken as a control sample.

2.2. Test procedures

For each cement, mortar was prepared using a well grade siliceous sand according to the ISO-RILEM guidelines and a cementitious material to sand ratio of 1:3. The water to cementitious material ratio (w/cm) was kept constant and equal to 0.50. Results of flow test (ASTM C 109) of studied mortars varied from 115% to 128% showing that both mineral additions or their combinations did not affect negatively the water demand of mortar. Mixtures were cast into 40×40×160 mm^3 prismatic molds and mechanically compacted in two layers. After casting, moulds were covered with a plastic sheet to prevent water loss and maintained in laboratory environment for 24 h. At this age, specimens were demolded and immersed in lime saturated water at 20±1 °C until test time.

At 2, 7, 14, 28, 90, 180 and 360 days, compressive strength was determined on pieces of prisms, according to ISO 679 standard, resulting from previous flexural strength test. Data reported represent the average values obtained from three flexural strength tests and six compressive strength tests.

After tests, fragments of mortar were ground to determine the amount of non-evaporable water (W_{ne}) according to the procedure proposed by Powers [15]. This value was used as a mean to estimate the progress of hydration reactions assuming that the LF is a hydraulically non-active component (it does not produce cementing compound) and all grains of BFS added are reactive.

2.3. Experimental plan

In this work, the experimental plan for determining the dependencies of strength with the percentage of LF and BFS was designed according to a two-level factorial plan. In order to include binary and ternary cements among experimental points, X_1–X_2 axes, representing the proportion of mineral additions, were rotated and transferred to the u–v axes as shown Fig. 1.

The quadratic response-surface model for these variables is:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_1^2 + \beta_4 X_2^2 + \beta_5 X_1 X_2$$

where Y is the property under evaluation (compressive or flexural strength), X_1 and X_2 are the experimental variables (X_1 is the percentage of LF and X_2 is the percentage of BFS), β_0–β_5 are the coefficients estimated using the least square method. All other controllable parameters (water-to-cementitious material—w/cm, cement/sand ratio, temperature, etc.) were kept constant.

As the location of the point representing the optimum combination of values for the experimental variables was unknown, it was appropriated to use a design giving estimations with equal precision in all directions. This property is called rotability and it can be assured by choosing the position of axial points $\alpha=(2k)^{1/4}$, where k represents the number of experimental variables under evaluation [16]. For this experimental design, there are two variables and then α is ±1.414.

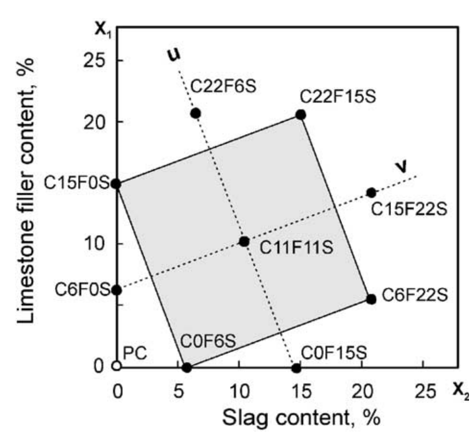

Fig. 1. Experimental design.

Table 2
Composition of blended cements

Mortar	Content (%)			Code values	
	Portland cement	Limestone filler (X_1)	Blast-furance slag (X_2)	u	v
PC	100	0	0	–	–
C0F6S	94	0	6	−1	−1
C0F15S	85	0	15	0	−1.414
C6F0S	94	6	0	−1.414	0
C15F0S	85	15	0	−1	1
C6F22S	72	6	22	1	−1
C11F11S	78	11	11	0	0
C15F22S	63	15	22	1.414	0
C22F6S	72	22	6	0	1.414
C22F15S	63	22	15	1	1

For the two level factorial plan, required mixtures (see Table 2) were: C11F11S corresponding to the central point (u=0 and v=0), four mixtures (C0F6S, C6F22S, C15F0S and C22F15S) corresponding to extreme levels for both experimental variables ($u=\pm 1$ and $v=\pm 1$) and four mixtures (C0F15S, C6F0S, C15F22S and C22F6S) corresponding to axial points ($u=\pm$ 1.414 and $v=\pm 1.414$). Finally, the number of mixtures tested in this experimental plan was nine (four binary cements and five ternary cements). Also, five central points (C11F11S mortar) were tested to obtain the uniform precision plan.

The coefficients of the quadratic response-surface for compressive and flexural strength were obtained for 2, 7, 14, 28, 90, 180 and 360 days. To determine β_0–β_5 coefficients according to orthogonal axis that defines the percentage of mineral additions (X_1 and X_2), the function obtained from the primitive axes (u and v) was rotated and transferred [17]. The coefficient of determination (R^2) measured the proportion of total variability in the response that is attributed to the model rather than to random error.

3. Results and discussion

Experimental results of compressive and flexural strength for each mixture are reported in Table 3. The β_0–β_5 coefficients obtained for the quadratic model for compressive and flexural strength at each test age are given in Table 4, where it can be observed that the coefficient of determination (R^2) was always equal or higher than 0.80, indicating a good correlation between calculated and experimental results. The maximum difference between the experimental and calculated compressive strength values was $\pm$6.4%, while this difference was $\pm$7.4% for flexural strength.

Results obtained from the experimental plan provide clear information about the contribution of each mineral addition or its combination to the strength (compressive/flexural) and the optimum replacement level for binary and ternary composite cements. Although PC mortar (X_1=0 and X_2=0) is not included in the experimental domain, this model also presented a good estimation of compressive and flexural strength with maximum error of 6.1% and 7.3%, respectively. In the model, the β_0 coefficient is the calculated value for PC mortar.

Figs. 2 and 3 illustrate the isoresponse curves for compressive and flexural strength, respectively. Analyzing the place where iso-lines cross the coordinate axes (X_1 and X_2), the contribution of each mineral addition (LF or BFS, respectively) on compressive strength can be easily observed. For instance, starting from 0 to the maximum on X_1 axis (Fig. 2a and b), it can be observed that incorporation of LF up to 15% produces an increase of compressive strength compared with experimental value of PC mortar at early ages (2 and 7 days). At 28 days (Fig. 2d), any iso-line crosses the X_1 axis from 0 to around 8% indicating a similar strength level compared to that obtained for PC mortar, but higher replacement levels produce lower compressive strength values. For later ages (Fig. 2e, f and g), the shape of the curves shows that compressive strength decreases when the percentage of LF increases in binary cement.

When BFS replacement is analyzed running from 0% to 22% on X_2 axis, the nature of the curves shows that compressive strength, compared with that of PC mortar, decreases when BFS content increases until the age of 28 days (Fig. 2a, b, c and d). After 90 days (Fig. 2e, f and g), there is no significant change of compressive strength for

Table 3
Compressive and flexural strength of cements

Mortar	Compressive strength (MPa)							Flexural strength (MPa)						
	2d	7d	14d	28d	90d	180d	360d	2d	7d	14d	28d	90d	180d	360d
PC	19.7	32.2	36.5	41.2	47.9	51.4	52.8	4.2	6.4	6.4	6.8	7.2	8.1	8.1
C0F6S	20.6	30.2	36.5	42.4	47.4	49.5	51.1	3.9	6.4	7.0	7.1	7.3	7.7	7.8
C0F15S	18.4	30.9	35.8	41.2	48.1	52.9	57.3	4.4	6.3	6.6	6.8	7.3	7.8	7.6
C6F0S	19.1	33.7	38.0	41.6	45.6	50.6	55.9	4.0	6.6	7.2	7.6	7.3	7.8	7.8
C15F0S	21.4	30.5	32.7	37.4	41.9	46.3	48.0	4.4	5.8	6.5	6.9	6.8	7.1	7.4
C6F22S	18.5	29.8	35.5	39.7	46.7	53.0	57.2	4.2	6.5	6.9	7.1	7.8	8.1	8.1
C11F11S	20.2	30.9	37.4	42.9	44.6	47.5	51.2	4.3	6.6	7.4	7.4	7.4	7.8	8.1
C15F22S	15.9	28.4	31.2	38.5	45.6	49.5	52.2	3.6	5.9	6.2	6.9	7.3	7.9	8.1
C22F6S	17.5	28.6	31.5	36.4	40.4	41.1	43.8	4.2	5.9	6.4	6.3	6.5	6.7	6.9
C22F15S	16.2	26.3	33.2	36.1	42.2	46.7	49.0	3.7	5.5	6.2	6.5	6.7	6.9	7.2

Table 4
Coefficients obtained for the quadratic model for compressive and flexural strength and coefficient of determination (R^2)

Age (days)	Coefficients β_0	β_1	β_2	β_3	β_4	β_5	R^2
Compressive strength							
2	18.51	37.06	19.92	−162.57	−105.63	−109.79	0.82
7	32.12	25.67	−3.41	−196.98	−47.08	−9.62	0.94
14	36.70	17.96	16.71	−205.30	−148.34	46.85	0.82
28	40.49	29.16	44.83	−305.78	−268.35	101.26	0.98
90	47.31	−51.35	−4.76	79.60	69.63	46.58	0.97
180	53.11	−55.40	−51.37	58.72	292.43	60.15	0.91
360	55.33	−21.51	−31.14	−117.55	211.42	43.47	0.83
Flexural strength							
2	3.81	5.28	5.66	−18.53	−21.70	−18.89	0.99
7	6.21	5.16	4.34	−39.14	−20.25	−3.17	0.80
14	6.41	12.24	10.07	−73.75	−54.60	7.66	0.80
28	7.20	9.01	2.65	−67.15	−15.23	17.70	0.91
90	7.20	9.01	2.65	−67.15	−15.23	17.70	0.91
180	7.67	4.18	0.41	−47.56	1.59	15.06	0.99
360	8.00	−2.37	−6.29	−16.25	27.74	26.03	0.97

mortars containing up to 14% of BFS, and it increases rapidly for high replacement level.

For ternary composite cements, it can be noticed that the parabolic isoresponse curve obtained for 2, 7, 14 and 28 days is characterized with a maximum compressive strength corresponding to an optimum percentage of LF and BFS. At 2 days, the maximum compressive strength (6% higher than the strength of PC) indicated by the stationary point (Fig. 2a) was obtained for the combination of 10% LF and 4% BFS. However, the zone delimited by the 20 MPa iso-line shows that the same strength level can be achieved by several LF/BFS combinations. The maximum value obtained is 20.8 MPa and it is located in the isoresponse zone including up to 18% LF and 14% BFS. Additionally, it is interesting to note that principal axes of this paraboloid are rotated indicating that strength depends on the sum of mineral addition replacement (X_1+X_2). At 7 and 14 days (Fig. 2b and c), isoresponse curves show a similar trend and the zone of maximum strength includes up to 17% LF–17% BFS and 11% LF–14% BFS, respectively. For 28 days (Fig. 2d), the stationary point is placed at 6% LF and 10% BFS. For 7, 14 and 28 days, the strength shows a marked decrease when LF content increases beyond 15% as can be noticed from the increment in iso-line density.

Since 90 days (Fig. 2e, f and g), the contour of isoresponse curves changes markedly and the maximum compressive strength corresponds to mortars with the largest percentage of BFS and a low percentage of LF (<5%).

The evolution of flexural strength shows a similar trend to compressive strength. The strength decreases for binary cements containing more than 14% LF after 2 days and there are not significant changes for binary cements containing BFS (none or one isoline crosses the X_2 axis). For ternary composite cements, the parabolic isoreponse curve shows a maximum of flexural strength up to 90 days. At early ages (Fig. 3a, b, c and d), this is obtained for LF replacements up to 17% and BFS contents up to 19%. As seen from Fig. 3f and g, for later ages flexural strength increases when BFS content increases and the highest value (8.2 MPa) was estimated for ternary mixture containing 8% LF and 22% BFS at 360 days.

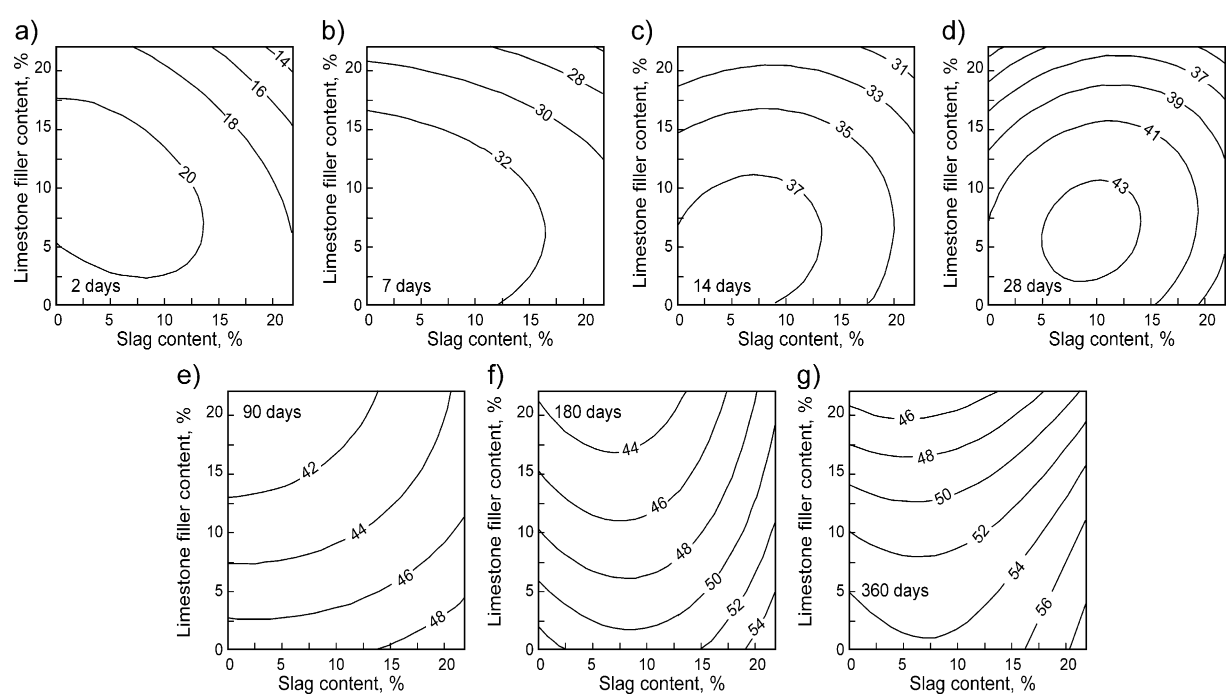

Fig. 2. Isoresponse curves for compressive strength.

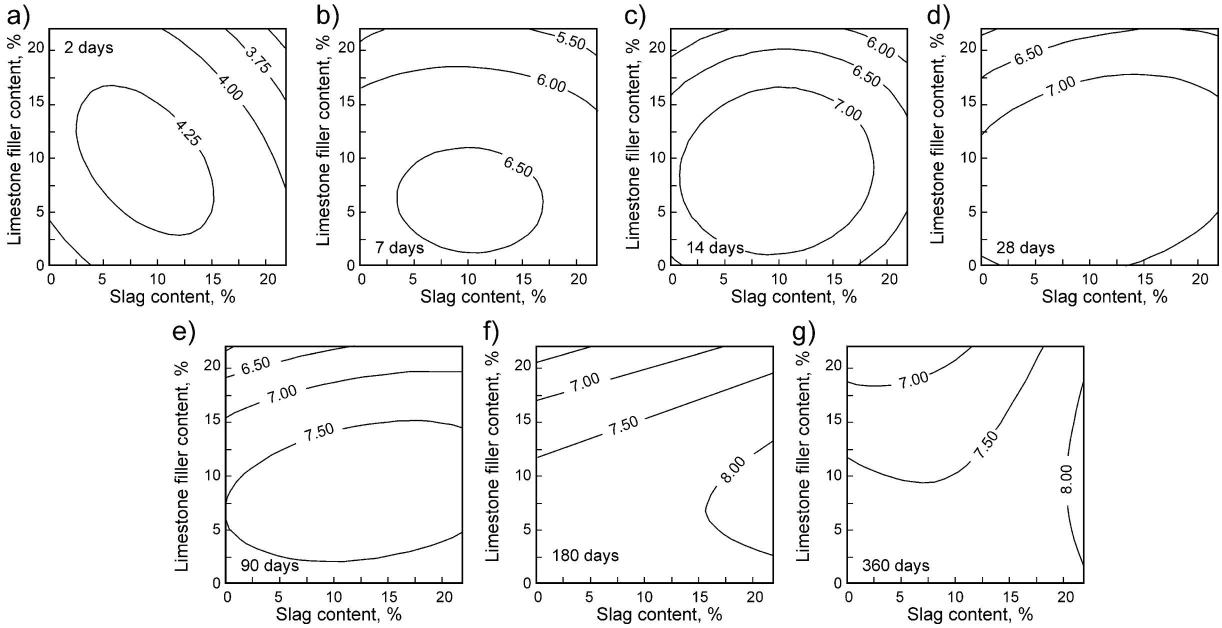

Fig. 3. Isoresponse curves for flexural strength.

In order to explain these results, the evolution of hydration for each mixture is analyzed. The increase of compressive strength in mortars containing LF compared with PC mortar up to 14 days can be attributed to the acceleration of hydration by the presence of fine particles of limestone filler. This increase of hydration is generally known as "filler effect", which yields to change the pore size distribution of mortar because the amount of smaller pores, compared to that of larger ones, increases with age more rapidly than for portland cement mortars [4]. In Fig. 4a, it can be observed that C6F0S and C15F0S mortars showed an increase of W_{ne} attaining values 26 and 30% higher than the corresponding to PC mortar, respectively.

After 28 days, W_{ne} of LF mortars was similar to PC mortar. However, its compressive strength presents a clear dependence on addition level that can be attributed to the reduction of potential cementing material, commonly called "dilution effect", causing a reduction of the volume of cementing hydrated material in the system. This strength reduction can be counteracted by a finer grinding of cement used to make the tailor-made cement [6].

For low percentages of BFS in binary cement (C0F6S), it can be observed that W_{ne} (Fig. 4a) was also higher than the corresponding to PC mortar at 2 and 7 days. According to Zhang et al. [18], BFS also produces the acceleration of hydration of clinker phases ("filler effect") at early ages. However, the rate of hydration of BFS is slower than that of the cement phase and, then it produces a smaller amount W_{ne} at 14 and 28 days causing a low strength mortar. In long-term hydration of BFS particles, hydration products formed during the reaction of the glassy structure of slag and the portland cement fraction are indicated by the increase of W_{ne} after 28 days (Fig. 4a). Thus, the system has the continuous ability to increase its amount of C–S–H and to attain added homogeneity and a fine dense microstructure by consumption of the crystalline CH released by

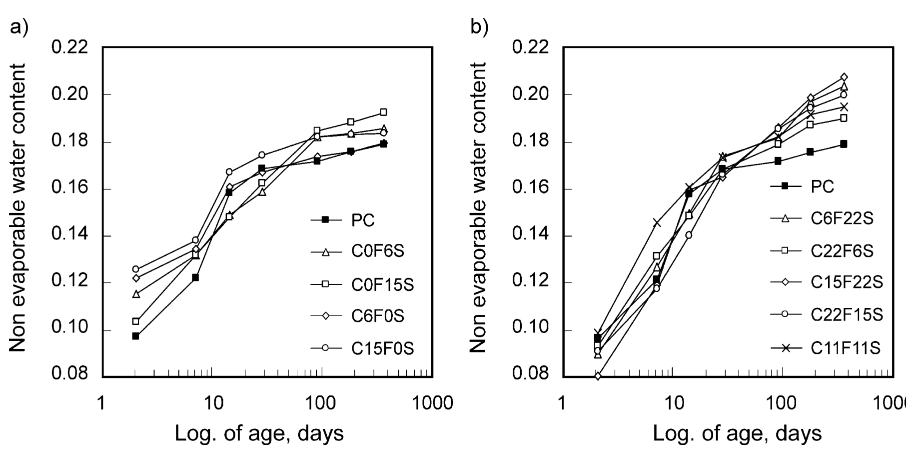

Fig. 4. Non-evaporable water content (W_{ne}) of: (a) binary cement mortars and (b) ternary cement mortars.

the silicate phases during hydration [19–21]. This process is called "pore-size refinement" and its rate depends on BFS particle size distribution. Generally, it is proposed that slag particles lesser than 10 μm contribute to early strengths up to 28 days, particles of 10 to 45 μm contribute to later strengths, but particles coarser than 45 μm results difficult to hydrate [1]. BFS used in the present work has about 50% of their particles between 10 and 45 μm and its contribution to strength development will be expected after 28 days. Thereafter, BFS hydration progress produces binary cement with higher strength at later ages.

Hydration of composite cements involving LF and BFS simultaneously is complex because it depends on the characteristics and percentage of mineral addition used [4]. For early ages (2 days), W_{ne} is similar to that of control cement with exception of C15F22S. At this age, it shows that W_{ne} decreases for mortars containing higher mineral addition percentages, especially for BFS (Fig. 4b). After 28 days, W_{ne} results higher for composite cements than for control cement and this effect is more noticeable for high BFS contents.

Mortar strength is also affected by the quality of the transition zone between the cement paste and the aggregates. This zone contains more voids, because of the difficulty of packing solid particles near a surface, and relatively more CH than elsewhere. These crystals grow large and tend to be strongly orientated parallel to the aggregate particle surface and, consequently, they are weaker than CSH and easily cleaved [22]. The incorporation of mineral additions, active or not, enhances the transition zone strength [23]. Hence, the improvement of flexural strength for composite cements could be attributed to two processes. One is the presence of very fine particles of mineral additions that causes an acceleration of cement phases hydration. The second effect is the "grain-size refinement" attributed to the nucleation of CH around the fine and well distributed particles of mineral additions that replace the large and oriented crystals of CH with numerous, small and less oriented crystals. When mineral addition is active, the formation of poorly crystalline reaction products in the cement matrix and the transition zone also will be computed [1,24].

Fig. 5 summarizes the optimum percentages of BFS and LF to obtain the highest compressive and flexural strength at different ages. At early ages, the maximum compressive strength is achieved for very low percentages of BFS and about 10% of LF, while at later ages the LF content should be reduced and BFS content increased. Regarding flexural strength, the optimum LF content remains around 10% up to 360 days, while BFS content changes from 9% at 2 days to 22% at 360 days. For all ages, it can be seen that there are composite cements with better response than the binary cement containing LF or BFS.

These results indicate that compressive and flexural strength are affected in different ways by the presence of mineral additions. In the first case, pore-size refinement plays a relevant role as can be inferred from increasing BFS contents necessary to achieve high strength at later ages.

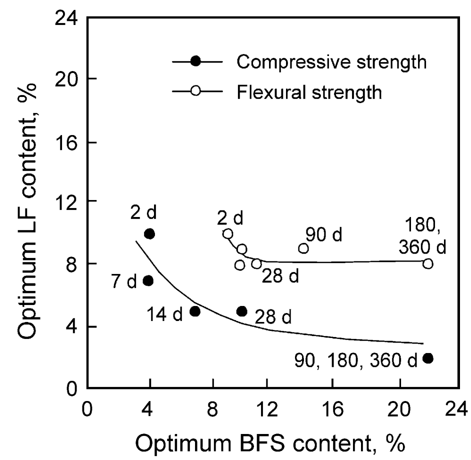

Fig. 5. Optimum percentages of BFS and LF for highest compressive and flexural strength at different ages.

However, flexural strength is affected to a greater extent by enhancement of transition zone and this is confirmed from optimum LF contents remaining almost constant from 2 to 360 days [4,22].

4. Conclusion

For the LF–BFS–PC system studied, containing up to 22% of LF and BFS, the following conclusions can be drawn:

- At all ages, there is a ternary blend of LF, BFS and PC that present an optimum strength, better than binary LF or BFS cement and plain portland cement. It is attributable to the complementary behavior of both admixtures: LF improves early strength while BFS improves later strength by its cementing reaction.
- The isoresponse method highlights the significance of the effect of the mineral addition and their interactions, and it permits to obtain the optimum combination to make a composite cement that meet with the standard or the user requirement regarding the environmental regulations (energy saving and emission reducing).

Acknowledgements

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