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Research article

Portland cement paste performance when inert limestone filler is added as clinker replacement

Andrés A. Torres-Acosta*, Rafael A. Méndez-Páramo, Celene Arista-Perrusquía, Eduardo S. Herrera-Sosa and Samantha Reyes-Rodríguez

Tecnologico de Monterrey, School of Engineering and Science, Department of Sustainable Technologies and Civil, Epigmenio González 500, San Pablo, 76130 Santiago de Querétaro, Qro., Mexico

* **Correspondence:** Email: atorresa@tec.mx; Tel: +52-81-8358-2000.

Abstract: Marginal information is available in the literature regarding the performance of Portland cement–based pastes when reducing clinker content with inert limestone fillers (ILSF) when the replacement is >15%. In this research, six different blended Portland cements (BPCs) from two different brands were evaluated, all with loss of ignition (LOI) values ranging between 2% and 18%, estimating ILSF contents between 5% and 35%. The characterization of these BPCs was conducted through laboratory tests aimed at performance forecasting in the absence of their component proportions due to trademark secrecy. These tests included determining BPCs' fineness (or Blaine number, F), density (ρ), and LOI. Then, paste characterization was performed, including water content for paste normal consistency, initial and final setting times, determination of the luminosity coordinate *L from hardened paste specimens, and mortar compressive strength at 28 days. It was concluded that one way to overcome the misinformation about the composition of BPCs (clinker, pozzolans, or ILSF contents) is to obtain the LOI parameter, as it will help forecast the performance of new BPC formulations.

Keywords: limestone; Portland cement; clinker; pastes; sustainability

1. Introduction

The design and construction of the new concrete road infrastructures (e.g., elevated viaducts, bridges, or docks) in Mexico have been affected by the increasing occurrence of early-age damage pathologies. These pathologies include the formation of fissures (<0.5 mm wide) and cracks (≥0.5 mm wide) due to accelerated drying or plastic shrinkage and the stains emanating from those cracks caused by chemical reaction leaching [1,2].

In many cases, this issue stems from a limited understanding of the long-term performance of hardened concrete manufactured with new formulations of Portland cement in infrastructure construction. Mainly, blended Portland cements (BPCs, as referred to in several countries regulations) are increasingly being used in construction in Mexico and several Latin American countries [3], replacing ordinary Portland cements (referred to as OPCs), which have a high clinker content (between 90% and 95%). In addition to the clinker content, other alternative products are added to BPCs, which, according to the Mexican Standard NMX-C-414-ONNCCE-2017 [4], may include granulated blast furnace slag (GBFS), natural pozzolans (NP), fly ash (FA), silica fume (SF), inert limestone filler (ILSF), and other minor components. These can be divided into two types: supplementary cementitious materials (SCMs; granulated blast furnace slag, natural pozzolans, fly ash, and silica fume) and ILSF. The first group reacts with water to generate hydration products, decreasing macro and capillary pores; the second group presents low or null reactions and only serves as filler material in the production of new BPCs.

Depending on the percentage content of these six components, six different types of cement can be defined, as outlined in the NMX-C-414-ONNCCE-2017 [4] Standard. Of these, the most commercialized is BPC; the other cements are manufactured on request, including OPCs. OPCs are a type of cement that was produced before 1999 in Mexico and other Latin American countries, having >90% clinker content based on the ASTM C-150 Standard [5]. However, this type of cement with a high clinker content is no longer produced in such countries. Only BPCs are marketed, and these may contain as little as 50% clinker [1,2].

In Latin America, modifications to cements since the late 20th century have followed a common strategy: reducing clinker to decrease CO_2 emissions and consequently mitigate the greenhouse gas (GHG) effects, which are becoming increasingly noticeable globally through various manifestations of climate change. At present, the reduction of GHG emissions through the decrease in clinker production in Portland cement is carried out by cement companies, transforming OPCs into BPCs. These reductions in clinker content are unknown to users of these new cements, creating doubts about their reactivity with chemical or mineral additives that could be used during the fabrication of the final products (mortar and concrete).

Table 1 shows the average clinker, pozzolans, and ILSF contents used in cements produced in Latin America from the 1990s to 2020 [3]. As observed from this table, gypsum content has remained relatively constant over the 30 years covered in the analysis. GBFS, FA, and NP increased throughout Latin America between 2000 and 2010. However, these decreased in the 2020s, apparently because the availability of these products declined due to transportation costs or production reduction [3].

This has allowed ILSF additions in Latin American cements to increase. Being the most readily available addition in all countries, because its quarries are located very close to cement factories, this makes it the most economical solution. Even in countries like Mexico, cement regulations allow for the addition of up to 35% ILSF to the binary or ternary mixtures of BPCs, a value much higher than what is allowed in other countries such as the United States and Canada [6]. To date, the Portland Cement Association (PCA) recommends the use of ILSF in concentrations between 5% and 15% [6]. Table 2 shows the results of a study sponsored by the PCA, illustrating the effect of ILSF content on the porosity and compressive strength of concrete [6].

Table 1. Averages of most common additions (in percentage) used in Latin American cements [3].

Year	SF	ILSF	GBFS	FA	NP	Other
1990	4	2	2	1	3	2
2000	4	5	4	2	6	2
2010	4	10	6	1	6	1
2020	4	13	4	1	4	1

SF: silica fume; ILSF: inert limestone filler; GBFS: granulated blast furnace slag; FA: fly ash; NP: natural pozzolans.

Table 2. Effect of limestone content in concrete [6].

Property	Porosity relative to OPC concrete	f´c (28 days) relative to OPC concrete
2%	0.9	1.10
5%	0.95	1.07
10%	1	1.02
15%	1.05	0.98

As observed in Table 2, the physical and mechanical performance of concrete improves when the cement has a low ILSF content (< 2%); concrete porosity is reduced by up to 10%, and its compressive strength at 28 days (f´c) increases by 10%. However, this performance improvement diminishes when ILSF addition occurs between 10% and 15%. Beyond 15%, the performance decreases: porosity increases by 15%, and f´c decreases by 10%. That is why the PCA limits the ILSF content to 15% as an addition to cement [6].

Such varied possible combinations for BPC addition content result in distinct performances, affecting their physical (porosity, plastic shrinkage), mechanical (compressive/tensile strength, modulus of elasticity/rupture), and durability (diffusivity/transport of aggressive agents within the concrete) performance [6]. This is why it is necessary to understand concrete performance if BPCs are used instead of OPCs to achieve sustainability that has been widely promoted in the concrete industry, especially in the cement industry. There has been an influx of information on social media and national and international newspapers claiming the achievement of a cement with “zero CO_2 emissions” and that this goal will be reached by 2030. However, the impact on the sustainability of concrete using these “zero CO_2 emissions” cements is unknown. Low clinker cement may be sustainable but with compromised concrete mechanical and durability performance [3].

ILSF is used in the same manufacturing process as clinker; its use is facilitated by it already being present in cement plants, with no need to transport additional raw materials, as would be the case with natural pozzolans (NP) or GBFS. Therefore, there would be no additional transportation costs or GHG emissions from the fuel used in transport.

Current Mexican regulations [4] allow the use of ILSF in Portland cements as the sole addition, maintaining the name of BPC (keeping users unaware of this particularity). Thus, from the standpoint of cement industry sustainability, the use of ILSF is the best option among all the additions accepted in Mexican regulations and those of many other Latin American countries.

2. Materials and methods

2.1. Materials

Considering three cement quality parameters (ρ, F, and LOI), an experimental program was planned with six different types of cement from two Mexican brands (for confidentiality reasons, the brands are not defined): BPC 20, BPC 30 R, BPC 30 R B, BPC 40, OPC 30 R SR LAAR, and BPC 30 R SR LAAR. Relevant information for each is listed below, and all cement's physical properties are listed in Table 3 (ND: not determined).

(1) BPC 20: this is a BPC-type cement with its main characteristic being a strength class of 20 MPa. This means that it achieves a compressive strength of 20 MPa at 28 days, obtained using standard mortar cubes according to ASTM C-109 [7]. The manufacturer provided the product in a bulk presentation. The last letter, F or H, corresponds to the cement's brand.

(2) BPC 30 R: a BPC-type cement with the main characteristic of having a strength class of 30 MPa; it is also designated as R because it achieves high strengths (>20 MPa) at a 3-day age. The manufacturer provided the product in a bulk presentation.

Table 3. Cement information obtained from the manufacturer's quality control reports.

Types of cement	ST_I (min)	ST_F (min)	LOI (%)	f^3_C (MPa)	f^{28}_C (MPa)	F (m^2/kg)
BPC 20 F	106	280	ND	18.4	26.6	575
BPC 30 R F	109	279	ND	25.3	36.1	520
BPC 30 R B F	102	233	ND	25.9	44.6	413
BPC 40 F	126	281	ND	34.8	46.1	466
OPC 30 RS LAAR H	173	327	3.1	22.8	40	337
BPC 30 R RS LAAR H	159	318	2.9	22.5	40.1	340

(1) BPC 30 R B: in addition to the characteristics of the previous cement (BPC 30 R), it is labeled B because it is a white cement. The manufacturer provided the product in a bulk presentation,

(2) BPC 40: a BPC-type cement with its main characteristic being a strength class of 40 MPa. The manufacturer provided the product in a bulk presentation.

(3) OPC 30 R SR LAAR: an OPC-type cement with a strength class of 30 MPa. The letter R represents its high strengths (>20 MPa) at three days of age; SR and LAAR represent the special characteristics of being sulfate-resistant and having low alkali-aggregate reactivity, respectively. The manufacturer provided the product in a bulk presentation.

(4) BPC 30 R SR LAAR: a BPC-type cement with its main characteristic being a strength class of 30 MPa; it is designated as R because it achieves high strengths (>20 MPa) at three days of age; SR and LAAR represent the special characteristics of being sulfate-resistant and having low alkali-aggregate reactivity, respectively. The manufacturer provided the product in a bulk presentation.

The types and proportions of the main elements of these cements (clinker, SCMs, ILSF) are not disclosed when purchasing the cement, only if the client asks for chemical composition (only oxide content, no phase content). Therefore, the information listed in Table 3 is the only known information for the six different cements evaluated in this investigation.

It is interesting to note that the information provided by the manufacturers of Cement F did not include LOI data, unlike Cement H. Another interesting point is that the fineness values of the evaluated cements (also called blaine) were in some cases >450 m^2/kg, indicating the high content of additions in these BPCs. It is important to clarify that the fineness value of clinker ranges between 350 and 400 m^2/kg, while that of inert limestone filler ranges between 600 and 1000 m^2/kg. This explains the high values obtained in the evaluated cements [8].

2.2. Cement characterization

These cements were characterized by the most common cement parameters: (1) density (ρ), following the standardized procedure of ASTM C-188 [9] or reference [3]; (2) fineness (F), following the standardized procedure of ASTM C-204 [10] or reference NMX-C-056-ONNCCE-2019 [11]; (3) LOI, following the standardized procedure of ASTM C-114 [12] or reference [3]; (4) amount of water for normal cement' consistency, using the standardized procedure of ASTM C-305 [13]; (5) setting times, using the standardized procedure of ASTM C-191 [14]; (6) cement paste luminosity index (*L), according to the standardized procedure of ASTM E-313 [15]; and (7) compressive strength, using 5 × 5 × 5 cm mortar cubes according to the standardized procedure of ASTM C-109 [7].

3. Results and discussion

The results obtained in this investigation for the BPCs are shown in Table 4. Values are averages based on three measurements, except for ρ, which is the average of two measurements per cement type. The results obtained in this research for each cement paste will be discussed below, directed toward laboratory tests that could provide an indication of the performance of each cement if the proportions of the main components (clinker, NP, FA, SF, GBFS, or ILSF) used by cement brands in their manufacturing are unknown.

Table 4. Average* physical characterization results of the evaluated cements.

Types of cement	w/c	ST_I (min)	ST_F (min)	ρ (g/cm^3)	LOI (%)
BPC 20 F	0.30	89	175	2.89	17.0
BPC 30 R F	0.29	110	195	2.91	11.5
BPC 30 R B F	0.28	100	200	3.05	6.8
PC 40 F	0.28	113	220	3.05	5.7
OPC 30 R RS LAAR H	0.26	125	250	3.13	3.3
BPC 30 R RS LAAR H	0.25	124	250	3.15	2.8

* Values are averages based on three measurements, except for ρ, which is the average of two measurements per cement type.

3.1. Cement powder characterization: ρ, F, and LOI estimates

Using the information regarding F (from Table 3, column 7) and ρ/LOI results listed in Table 4 (columns 5 and 6, respectively), Figures 1–3 were generated to relate the strength class of the cement to its properties. It should be noted that unfilled circles (○) correspond to all BPC types, and black-filled circles (●) represent OPC, being considered the control cement.

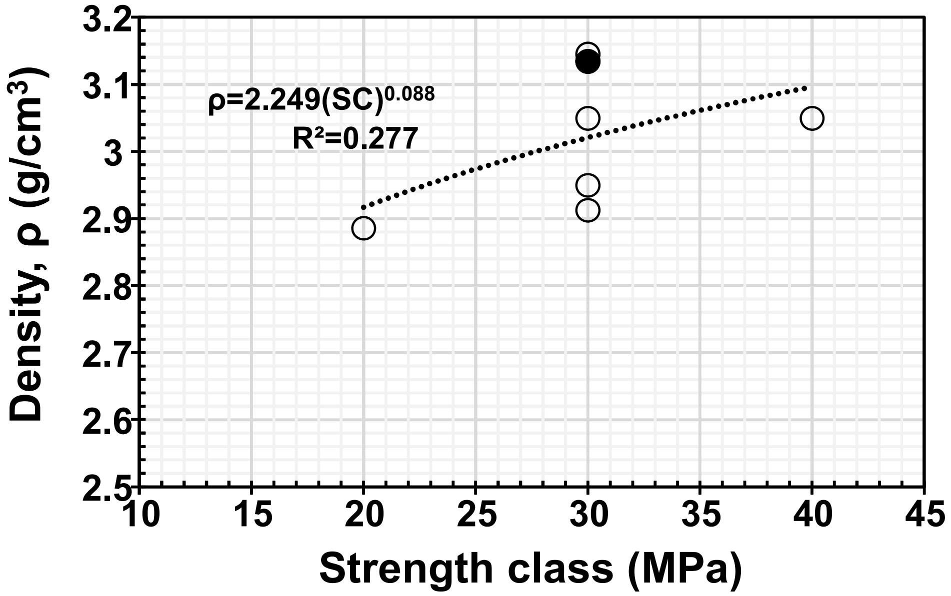

Figure 1. Average cements' density (ρ) vs. strength class. (●) OPC; (○) BPC.

As demonstrated by Figure 2, the strength class 20 cement achieved the lowest ρ and the highest F and LOI values among all evaluated cements. On the contrary, strength class 40 cements showed the highest ρ and lowest F and LOI.

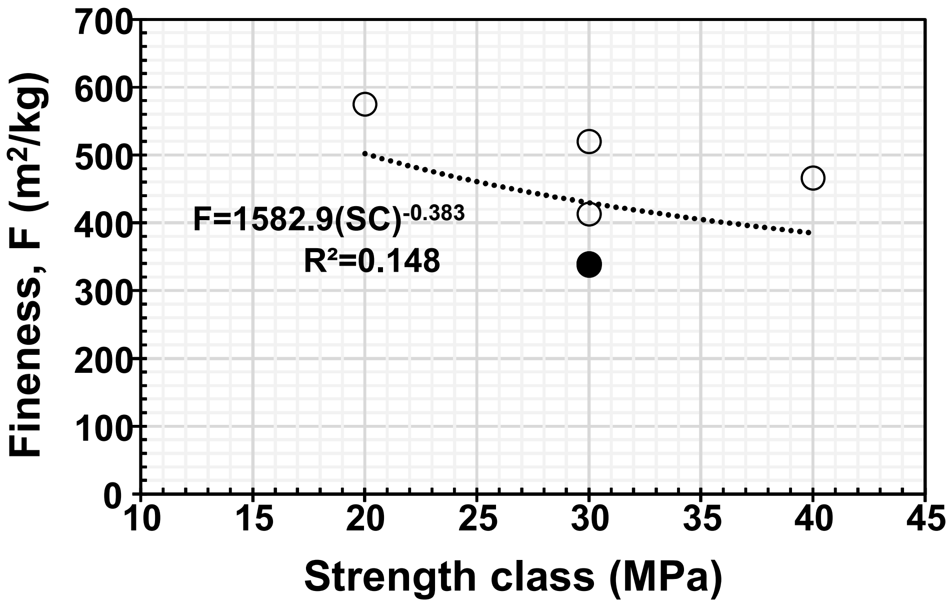

Figure 2. Average cements' fineness (F) vs. strength class. (●) OPC; (○) BPC.

However, when comparing cements of the same strength class (namely class 30, as only one cement was available for evaluation for classes 20 and 40), type BPC 30 R RS BRA and type OPC 30 R RS BRA achieved the highest ρ and the lowest F and LOI for this strength class. BPC 30 B and BPC 40 had very similar ρ values (Table 4), while LOI values differed by one unit only, implying a reduction of inert material in type BPC 40 compared to type BPC 30 B, which aligns with the designation of their strength class.

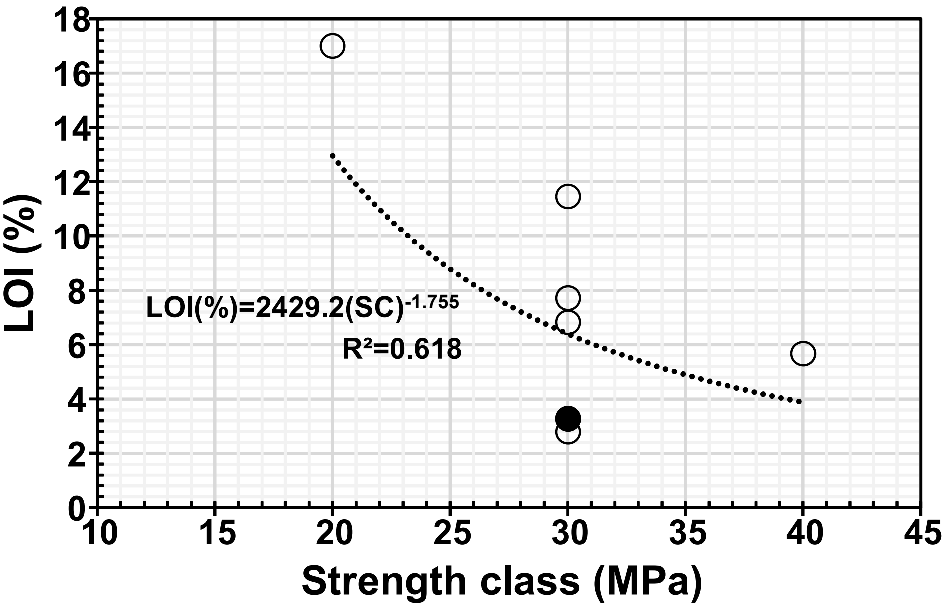

Figure 3. Average cements' loss of ignition (LOI) vs. strength class. (●) OPC; (○) BPC.

Figure 4 was generated using the values obtained for ρ and LOI, showing an excellent correlation between both parameters: the higher the LOI value, the lower the ρ of the cement.

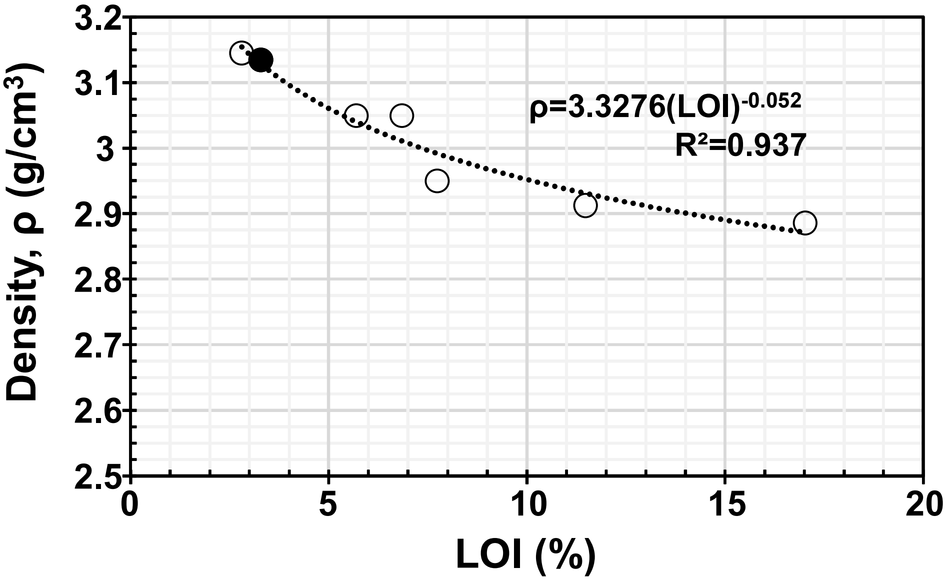

Figure 4. Average cement's density (ρ) vs. loss of ignition (LOI). (●) OPC; (○) BPC.

This would suggest that if the concrete mix design method according to ACI 211 [16] is used, the actual ρ of the cement should be estimated for determining the other mix design components, since the current value of 3.15 g/cm^3 corresponds to OPCs' ρ, which is composed only by clinker and gypsum.

Therefore, greater importance should be given to the determination of cement's ρ and LOI estimates, so that the mix design of these concretes is not affected by the type of cement used.

The real ρ value will help estimate the actual volumes of the other components (primarily aggregates), which will be proportioned for the project. If a cement with a lower ρ than the theoretical 3.15 g/cm^3 is used, errors in proportioning may occur, and consequently, the specified strengths may not be achieved. Similarly, lack of knowledge regarding the clinker content in the cement will also affect its mechanical strength, as it is related to the water/clinker (w/cl) ratio and not to the water/cement ratio if the cement has pozzolans or ILSF additions. Therefore, it is necessary to change the way BPC performance is assessed, using new testing procedures that can be extrapolated to materials where these new BPCs are used.

Using the values of F reported by the manufacturers and ρ/LOI estimates obtained here for all nine cements, Figures 5 and 6 present the plots of F vs. ρ and F vs. LOI, respectively. As observed from both figures, excellent correlations were obtained between these three parameters: the higher the F, the lower the ρ, and the higher the LOI.

From all previous data correlations, it is interesting to observe that all six bulk-type cements evaluated follow the same trends; thus, an apparent conclusion is that these six cements have only ILSF as the main addition.

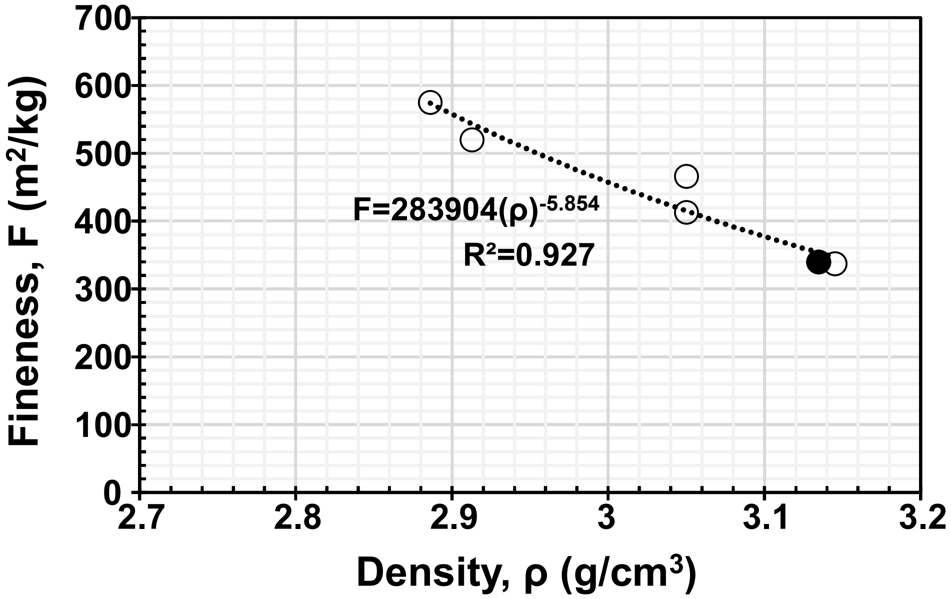

Figure 5. F vs. ρ. (●) OPC; (○) BPC.

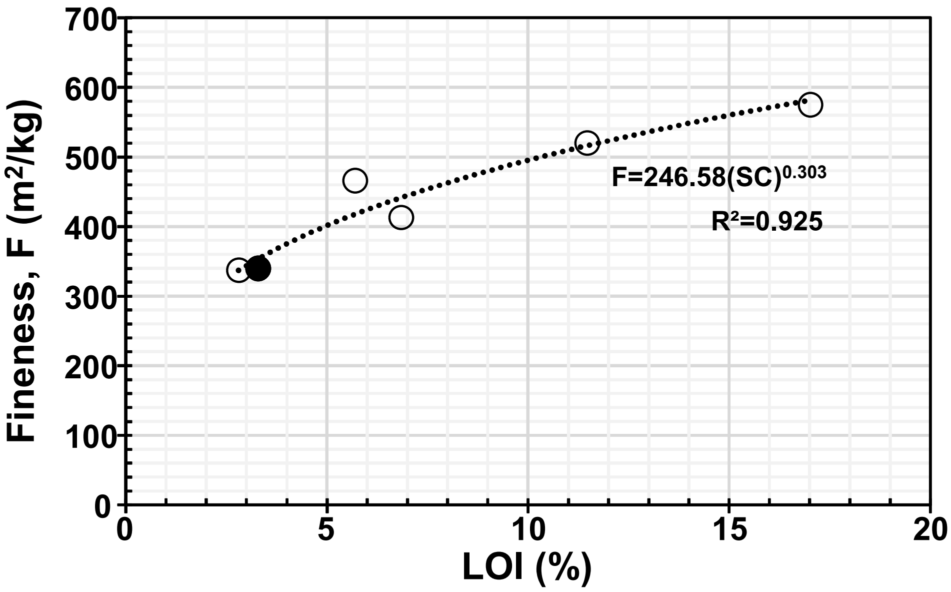

Figure 6. F vs. LOI. (●) OPC; (○) BPC.

3.2. Cement paste's water content for normal consistency

The second column of Table 4 shows the average values of the water/cement (w/c) ratio obtained for the six cements evaluated to achieve a normal paste consistency, as defined in the procedure from Standard ASTM C-305 [13]. Using these w/c values from Table 4 and the compressive strength class of the evaluated cements, Figure 7 was generated. It shows a fair correlation between both parameters, where a lower compressive strength class (for example, 20) corresponds to a higher w/c to achieve normal consistency in the paste. This implies that these cements have a higher addition content in their formulation and, therefore, a lower clinker content, causing pastes manufactured with these high-addition cements to exhibit diverse hydration reaction performances, which will be evaluated in the following sections of this investigation (setting times determination).

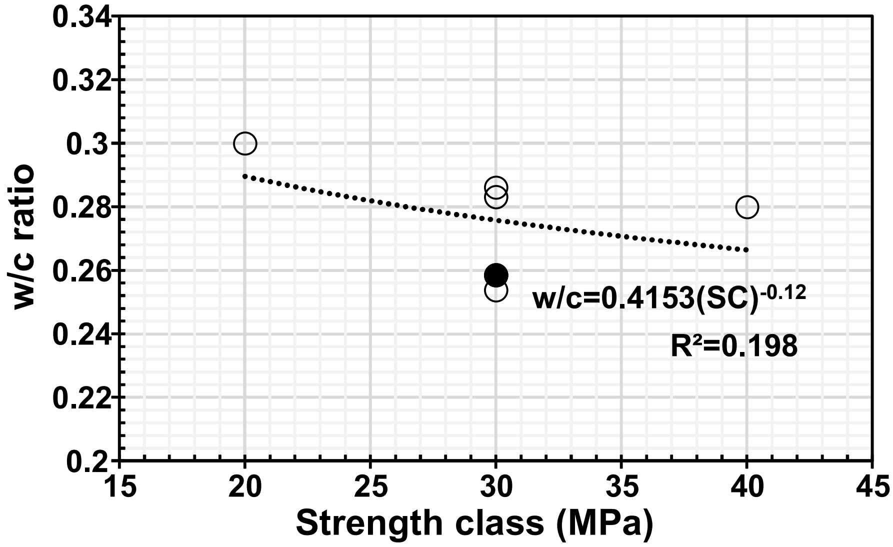

Figure 7. Average w/c to obtain normal paste consistency vs. strength class. (●) OPC; (○) BPC.

Based on the ρ and w/c values obtained and listed in Table 4 (column 2), Figure 8 was plotted. As observed in this figure, the amount of water needed to achieve a paste with normal consistency decreases as the ρ decreases too. The lowest w/c obtained was for the OPC paste, indicating, again, that if ILSF or other addition replaces clinker, the need for more water to cover the entire composite cement is unavoidable. Therefore, pastes will have increased porosity due to the water in excess needed to achieve such normal consistency.

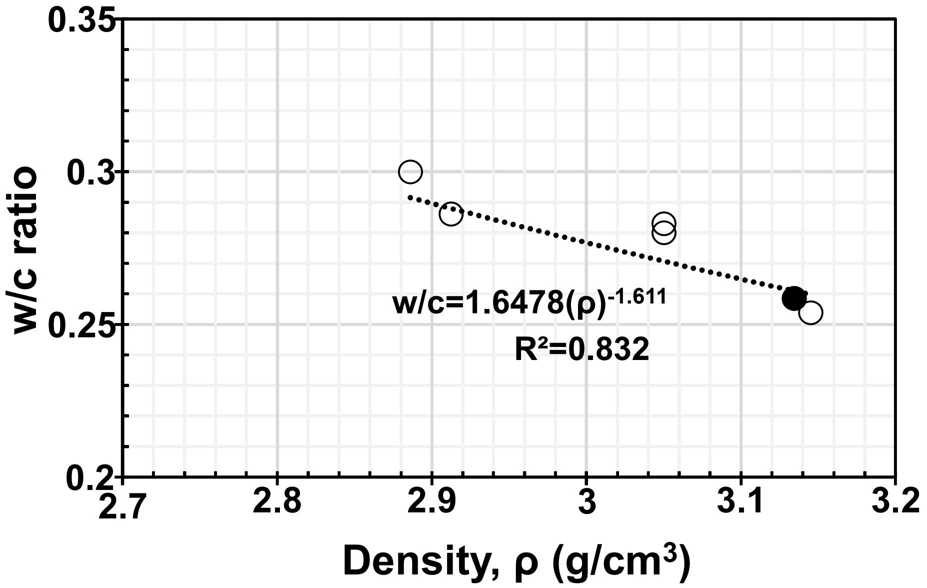

Figure 8. Average w/c to get a normal paste consistency vs. ρ. (●) OPC; (○) BPC.

Using these w/c results and the LOI values from Table 4 (column 6), Figure 9 was plotted. The results show that as the LOI value increases, indicating a higher ILSF content, the cement paste requires more water to achieve the same normal consistency according to the standard ASTM C-305 method [13]. This implies that if the ILSF content in the cement is high, a greater amount of water will be needed to achieve the same paste workability. This increase in water implies that pastes with cements having higher LOI will have a higher water/cementitious (w/cm) ratio than Portland cements containing only clinker and gypsum. In other words, the paste compressive strength with high ILSF additions (or higher LOI), will be lower than that of OPC without these additions.

Using the w/c results in Table 4 (column 2) and the F values reported by the manufacturers and listed in Table 3 (column 7), Figure 10 was plotted. Again, an excellent correlation was observed between these two paste parameters: the higher the F, the more water is needed to achieve normal consistency in the paste.

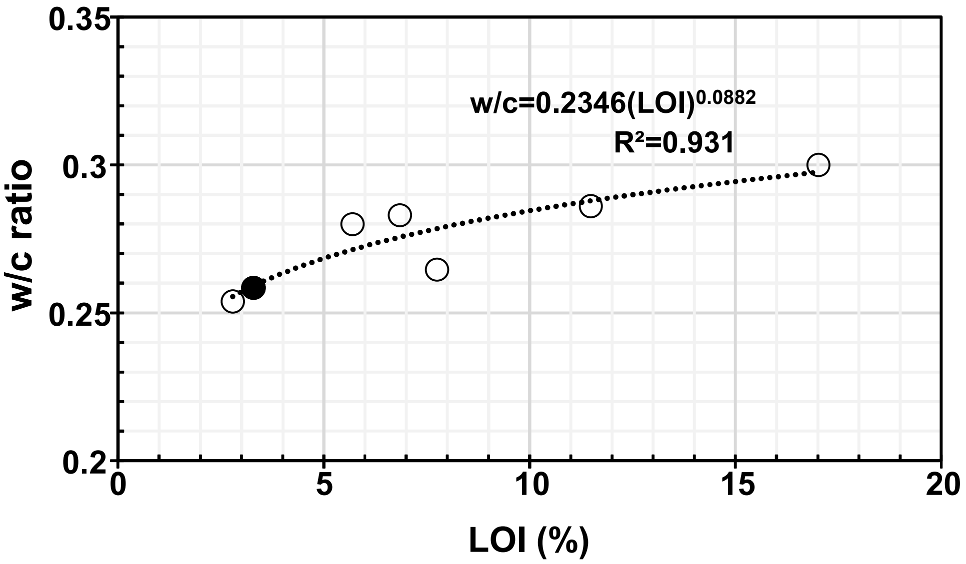

Figure 9. Average w/c to obtain a normal paste consistency vs. LOI. (●) OPC; (○) BPC.

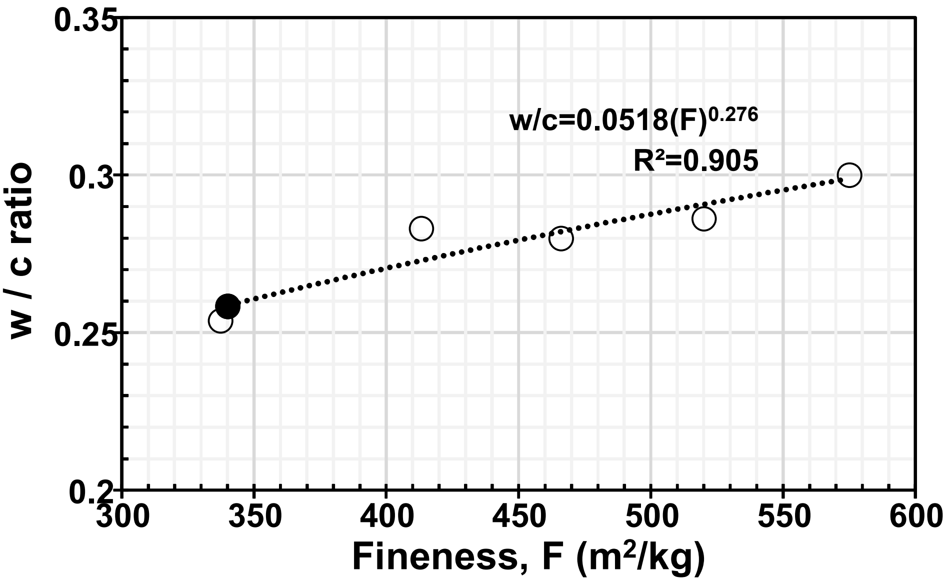

Figure 10. Average w/c to obtain a normal paste consistency vs. F. (●) OPC; (○) BPC.

It is also important to mention that this increase in the amount of water for either low ρ or high F/LOI BPCs should be analyzed in more detail to plan the modification of ASTM C-305 [13] standard, where the water estimated by this method reacted entirely with the cement to obtain normal consistency when the clinker content is high. For cements with low clinker contents (<85%), a significant portion of the water will not react with clinker. This means that pastes made with these BPCs could be used with the water/cementitious material (w/cm) ratio instead of the w/c ratio, which has been used by the ASTM C-305 Standard [13] regularly since its creation in the early 20th century, when additions were not included in Portland cement formulations.

In other words, a modification to the ASTM C-305 Standard [13] should be considered to fix a constant w/cl ratio, and the normal consistency of pastes should be achieved with varied water-reducing chemical admixture until reaching this normal consistency of 10 mm penetration of the Vicat apparatus [13]. This will remain an idea and, later, when more information is available, a modification to the Standard ASTM C-305 [13] needs to be considered.

3.3. Cement paste setting times

Initial and final paste setting times (ST_I and ST_F, respectively) are listed in columns 3 and 4 of Table 3. These estimates are plotted against the strength class in Figure 11. It can be observed that both ST_I and ST_F are affected by the type of cement, which is differentiated by their strength class in the figure.

A good correlation is observed between this strength class and the ST_I and ST_F of the evaluated cement pastes: the lower the strength class, the smaller the ST_I and ST_F. This implies that lower strength class cements must contain higher levels of additions that accelerate the setting process. It is known that ILSF-based additions produce this effect of accelerating the setting or hardening of Portland cements, which suggests that most of the evaluated cements only have this addition.

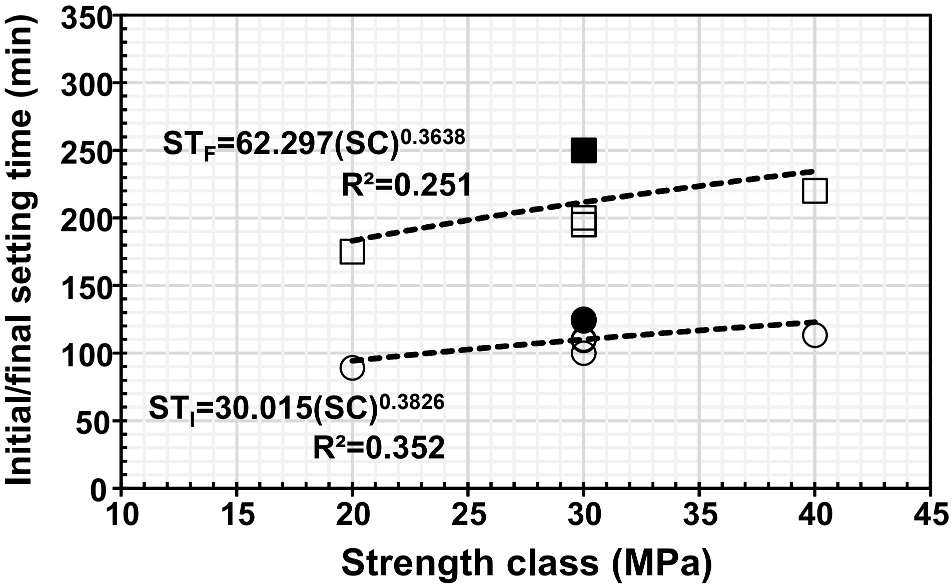

Figure 11. Average initial/final setting times (ST_I/ST_F) of paste vs. strength class. (●,■) OPC; (○,□) BPC.

Figure 12 shows the ST_I and ST_F as a function of LOI. As observed in this figure, these STs decrease as BPC's ρ decrease, or both F and LOI values increase. It has been discussed in previous research that ILSF reacts with water to form solid carbonates, which can reduce porosity [6]. However, this reaction occurs with a very small ILSF percentage (<2%), leaving a significant amount of this inert material unreacted. This unreacted material absorbs water from the mix, causing the cement paste to harden more quickly [6].

Once again, the diverse performance of the different cements evaluated in this research alerts users, as they will generate different performances in the materials where these cements are used (mortars and concretes). This is why knowing clinker and addition contents in these so-called blended cements is necessary: to control potential detrimental effects on mortars and concrete using them as binders for their fabrication.

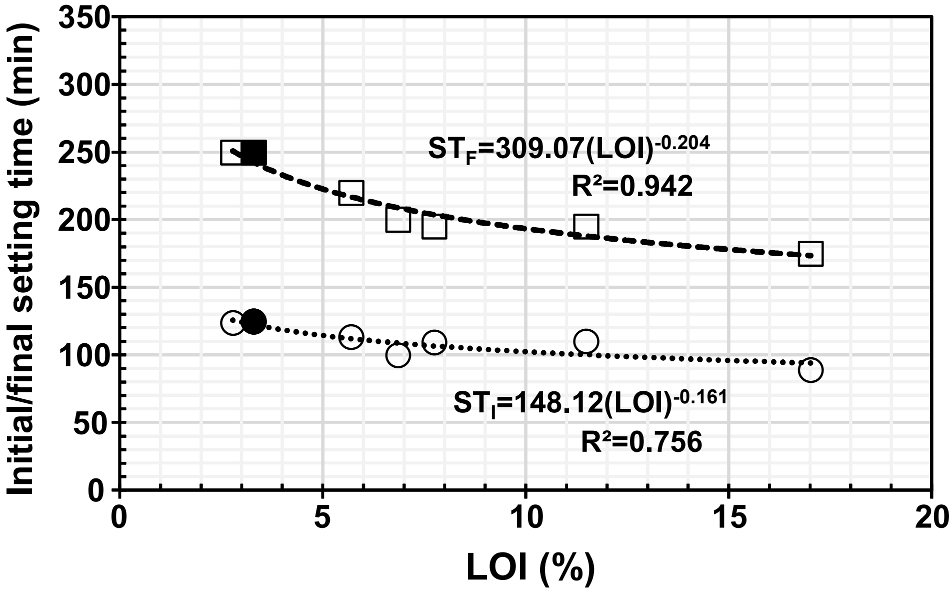

Figure 12. Average initial/final setting times (ST_I/ST_F) of paste vs. LOI. (●,■) OPC; (○,□) BPC.

Just as discussed in the previous section regarding a potential modification to the Standard ASTM C-305 [13], it is also necessary to consider the possibility of amending Standard ASTM C-191[14]. This modification would involve estimating the initial and final setting times using a constant w/cm ratio and the quantity of chemical water-reducing admixture needed to achieve normal consistency. Subsequently, pastes would be prepared with these proportions to assess whether the setting times remain unchanged. This suggestion is put forward for future short-term work, allowing for the observation of the performance of pastes with the same amount of water as if it were ordinary Portland cement.

3.4. Cement pastes' luminosity index

Following paste specimens manufacturing for setting, they were stored in bags to maintain humidity for 28–30 days. Once this curing time was over, they were left in dry bags so that they lost their humidity for a period of 90 days inside the bags to avoid carbonation.

Once the cement paste specimens were dry, a colorimetry test was carried out using a colorimeter using the standardized procedure of ASTM E-313 [15]. This equipment determines the three coordinates of the CIE color space L*, a*, and b*. The L*a*b* color space, also referred to as CIELAB, is currently one of the most popular and consistent color spaces used to evaluate the color of an object. This color space is widely used because it correlates numerical color values consistently with human visual perception.

The L*a*b* color space was modeled based on an opponent color theory that states that two colors cannot be red and green or yellow and blue at the same time. L* indicates the luminosity and a* and b* are the chromatic coordinates, defined as follows: L* = luminosity, a* = red/green coordinates (+a indicates red, −a indicates green), and b* = yellow/blue coordinates (+b indicates yellow, −b indicates blue).

As an example, the theoretical “perfect white” has reflectance values of 100% across the visible spectrum with corresponding colorimetric values of L* = 100.00, a* = 0.00, and b* = 0.00. If an item is almost, but not perfectly, white, it may be darker (has a lower L* value) and possibly be slightly chromatic, either in the red-green dimension (a*) or in the yellow-blue dimension (b*).

In the case of the evaluated cement pastes, a colorimeter was used to obtain these three chromatic coordinates to determine if they can be related to the amount of ILSF, since the powder of this product has a more intense white than the color of clinker or many other SCMs. Table 5 lists the average values measured in each of the cement paste specimens used to determine the TIF and TFF. Three measurements were made per specimen, and the number of specimens was between 2 and 3, so the average L*, a*, and b* values were estimated to be between 6 and 9 measurements.

Table 5. Average CIELAB space coordinate results for the evaluated pastes (average of 6–9 measurements).

Types of cement	L*	a*	b*
BPC 20 F	67.23	0.97	4.02
BPC 30 R F	59.03	0.26	3.84
BPC 30 R B F	91.84	−0.07	3.87
BPC 40 F	61.84	0.33	2.99
CPO 30 R RS BRA H	51.84	−0.52	2.93
BPC 30 R RS BRA H	52.61	−0.06	4.69

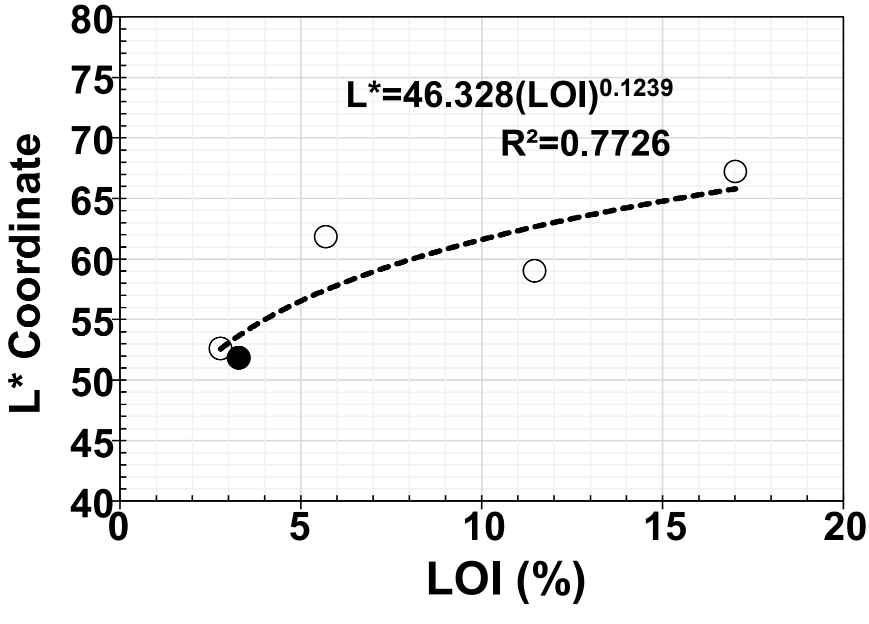

Figure 13. Average pastes’ L* coordinate vs. LOI. (●) OPC; (○) BPC.

Because the L* coordinate helps define the whiteness of the material, the average values of this coordinate were plotted as a function of the LOI for each cement, as shown in Figure 13. The value obtained from BPC 30 R B is not considered in Figure 13, since it is a white cement, and its value does not reflect the ILSF content only. Figure 13 shows a good correlation of both parameters (regression coefficient R^2 = 0.773), which implies that this test could also help determine the ILSF content in cements to control their quality.

3.5. Mortar's mechanical compressive strength at 3 and 28 days of curing

As additional information, mortar cubes (5 × 5 × 5 cm) were manufactured to obtain the compressive strength of the mortar after 3 (f^3_C) and 28 days (f^{28}_C) of curing, following the procedure of the ASTM C-109 standard [7]. As a reminder, the strength class is the compressive strength of each cement from mortar cube testing at 28 days of curing. However, if the cement has, in addition, an "R" in its nomenclature, it means that the cement may reach an early age strength of 20 or 30 MPa at 3 days of curing if the strength class is 30 or 40, respectively. Of the six cements evaluated in this investigation, four of them were defined by the manufacturers as strength class R (BPC 30 R, BPC 30 R B, OPC 30 R RS LAAR, and BPC 30 R RS LAAR).

The average results (from three cubes) listed in Table 6 (columns 2 and 3) for each mortar mixture are presented in Figure 14 for f^3_C (○ symbols) and for f^{28}_C (□ symbols), respectively, vs. the strength class defined by the manufacturers of the evaluated BPCs. Data is also compared with the 1:1 discontinuous line to determine possible differences between each cement's strength class and the estimated strength for both curing times. From the f^3_C average results shown in Figure 14 (○ symbols), two out of four BPC 30 R did not reach the 20 MPa needed for being assigned as strength class R. The best f^3_C performance was for the CPO 30 R SR LAAR cement, which reached an average of 23.4 MPa at 3 days.

The average f^{28}_C vs. strength class data plotted in Figure 14 (□ symbols) also shows a good approximation to the 1:1 line for strength classes 20 and 40. For strength class 30, only one cement type (BPC 30 R) was below the 1:1 line; the others were above. One of the cements evaluated (BPC 30 B) even reached f^{28}_C = 40 MPa, which reached 33% higher strength than the specified. However, as observed in Figure 14, most cement types reached the strength class defined by the manufacturers regardless of the addition type or amount present in the BPC formulation.

Table 6. Average compressive strength from mortar cubes, at 3 (f^3_C) and 28 days (f^{28}_C) of curing, for the evaluated cements (average of three cubes).

Types of cement	f^3_C (MPa)	f^{28}_C (MPa)
BPC 20 F	15.7	19.4
BPC 30 R F	16.9	23.3
BPC 30 R B F	22.9	39.9
BPC 40 F	26.3	39.4
OPC 30 R RS LAAR H	16.3	31.2
BPC 30 R RS LAAR H	23.4	31.0

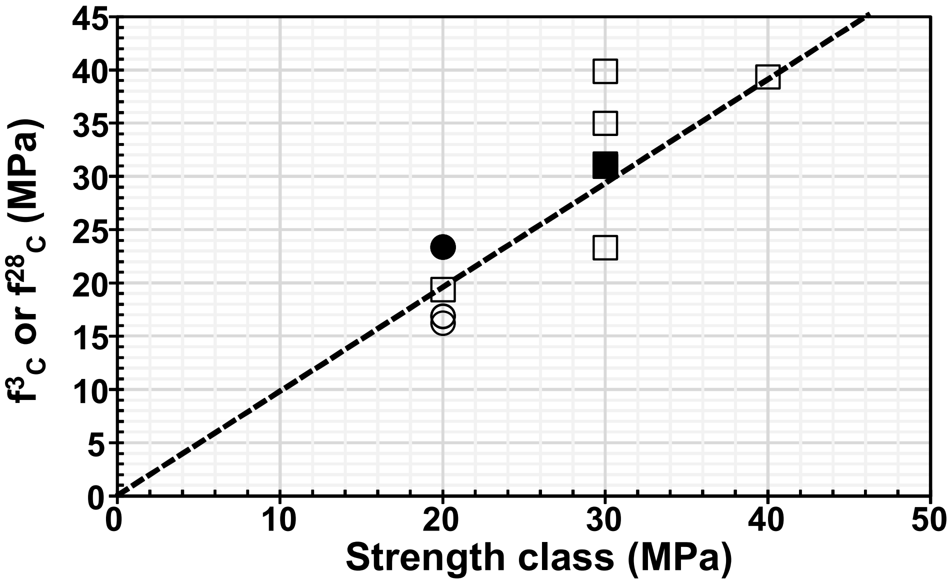

Figure 14. Average mortars' f^3_C (● OPC; ○ typical BPC) and f^{28}_C (■ OPC; □ typical BPC) estimates vs. strength class.

Correlating the average values of f^3_C or f^{28}_C and LOI, Figure 15 was prepared using data from Table 4, column 6, and Table 5, columns 2 and 3. As observed from Figure 15, there is not an apparent correlation between cement's LOI and mortar's f^3_C (Figure 15a, $R^2 = 0.117$) or f^{28}_C (Figure 15b, $R^2 = 0.265$). These correlations need to be further evaluated with more experimental results to continue the evaluation of other cement types.

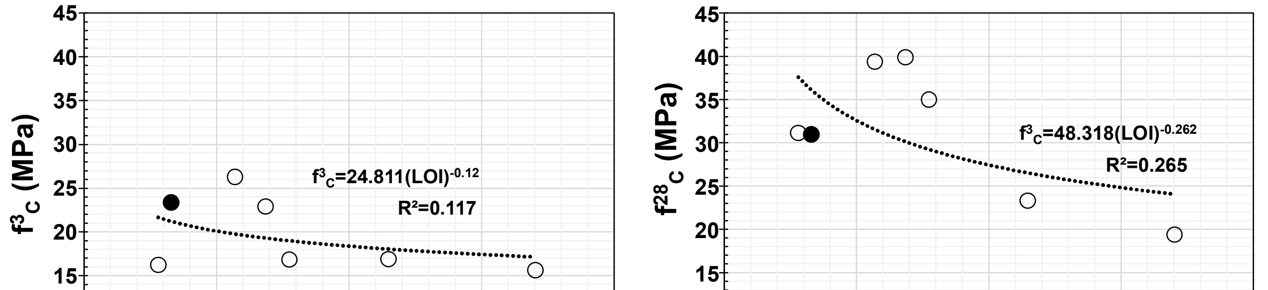

Figure 15. Average mortars' f^3_C and f^{28}_C vs. LOI. (●) OPC; (○) BPC.

With these observed differences, certain precautions should be taken when using BPCs without knowing their basic physical characteristics (ρ, F, LOI, ST_I, and ST_F). Therefore, users should be aware of these characteristics to better design their mixes and ensure that their concrete meets the required specifications.

3.6. Empirical correlation to forecast w/c, ST_I/ST_F, and f^{28}_C from ρ, F, and LOI

With the results obtained for ρ, F, and LOI for the six cement types evaluated, a multiple regression was performed to obtain the amount of water (from the w/c ratio) needed to get normal consistency and the ST_I/ST_F estimates for each paste.

This empirical equation was obtained using the multiple linear regression tool provided by Excel. The procedure used the average ρ, F, and LOI from each cement type, which is listed in Table 6, along with its w/c ratio, ST_I, and ST_F value. Being a linear regression tool, the base-ten logarithms of the experimental values were estimated to perform this regression using Eqs 1–4:

$$\text{Log } w/c = A_1 \text{ Log }(\rho) + B_1 \text{ Log }(F) + C_1 \text{ Log }(LOI) + D_1 \quad (1)$$

$$\text{Log } ST_I = A_2 \text{ Log }(\rho) + B_2 \text{ Log }(F) + C_2 \text{ Log }(LOI) + D_2 \quad (2)$$

$$\text{Log } ST_F = A_3 \text{ Log }(\rho) + B_3 \text{ Log }(F) + C_3 \text{ Log }(LOI) + D_3 \quad (3)$$

$$\text{Log } f^{28}_C = A_4 \text{ Log }(\rho) + B_4 \text{ Log }(F) + C_4 \text{ Log }(LOI) + D_4 \quad (4)$$

Once the values of constants A–D of each parameter used as a variable were obtained, the regression generated the following linear equation (Eqs 5–8) (rounded to three decimal places):

$$\text{Log } ST_I = -7.808 \text{ Log }(\rho) + 0.108 \text{ Log }(F) - 0.593 \text{ Log }(LOI) + 5.991 \quad (5)$$

$$\text{Log } ST_F = -3.163 \text{ Log }(\rho) + 0.069 \text{ Log }(F) - 0.383 \text{ Log }(LOI) + 3.977 \quad (6)$$

$$\text{Log } w/c = 2.442 \text{ Log }(\rho) + 0.180 \text{ Log }(F) + 0.158 \text{ Log }(LOI) - 2.335 \quad (7)$$

$$\text{Log } f^{28}_C = 27.195 \text{ Log }(\rho) + 1.742 \text{ Log }(F) + 0.596 \text{ Log }(LOI) - 16.686 \quad (8)$$

The four empirical equations were obtained by transforming them into a series of multipliers whose sign would define whether they were placed in the numerator or denominator of the equation. The factors of the empirical equations are ordered using the rule of logarithms, giving Eqs 9–12:

$$ST_I = \frac{9.795 \times 10^5 \times F^{0.108}}{\rho^{7.808} \times LOI^{0.593}} \quad (9)$$

$$ST_F = \frac{9.484 \times 10^3 \times F^{0.069}}{\rho^{3.163} \times LOI^{0.383}} \quad (10)$$

$$\frac{w}{c} = 4.624 \times 10^{-3} \times \rho^{2.442} \times F^{0.18} \times LOI^{0.158} \quad (11)$$

$$f^{28}{}_C = 2.061 \times 10^{-17} \times \rho^{27.195} \times F^{1.742} \times LOI^{0.596} \quad (12)$$

Figure 16 compares the experimental value for ST_I and ST_F with the estimated ones using Eqs 9 and 10. Figure 17 compares the experimental value for the w/c ratio and f^{28}_C with the estimated ones using Eqs 11 and 12. The same figures show the correlation equations between both experimental and estimated values and the corresponding correlation coefficients R^2.

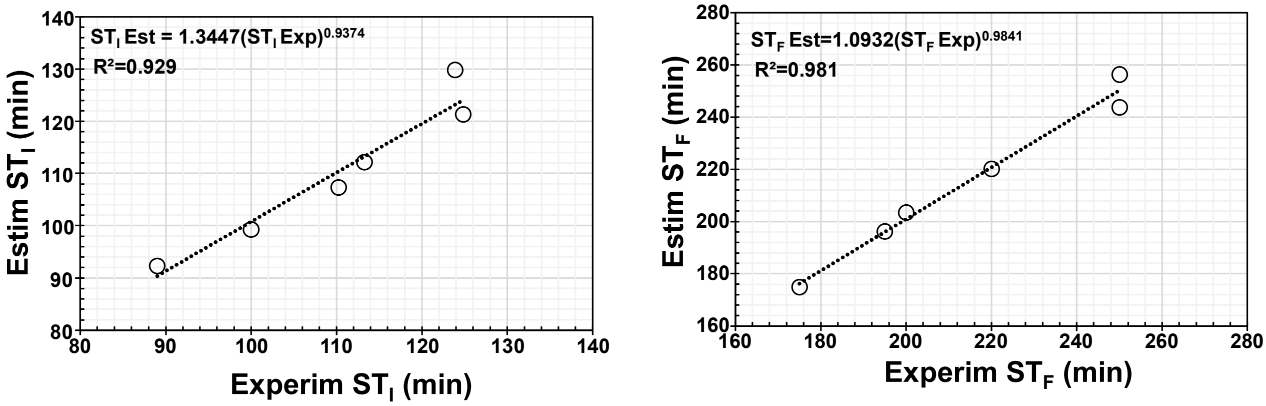

Figure 16. Experimental vs. estimated values of (a) ST_I and (b) ST_F. Estimated values for ST_I and ST_F were obtained with Eqs 9 and 10, respectively.

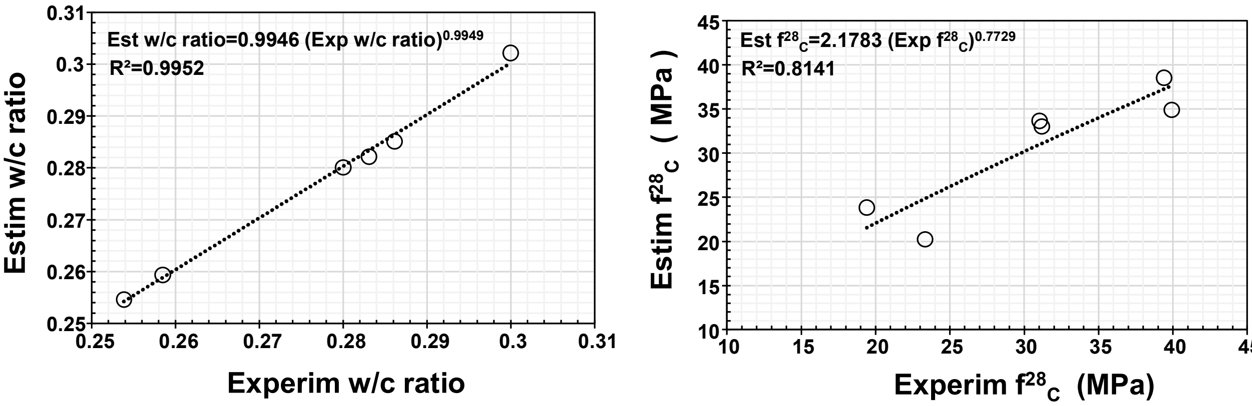

Figure 17. Experimental vs. estimated values of (a) amount of water for normal consistency, represented as w/c ratio, and (b) $f^{28}{}_C$. Estimated values for the w/c ratio and $f^{28}{}_C$ were obtained with Eqs 11 and 12, respectively.

As can be seen, the R^2 coefficient was relatively high for experimental vs. estimated ST_I/ST_F (0.929 and 0.9813 for ST_I and ST_F in Figure 16a,b, respectively) and experimental vs. estimated w/c ratio (0.9952) (Figure 17a). The tendency lines were very close to the equity line for these three experimental vs. estimated graphs (Figures 16a,b, and 17a) of the form Estim = A(Experim)^n, where A ~ 1.0 and n ~ 1. This suggests that the first steps of cement hydration could be forecasted with the powder properties of fineness (F), density (ρ), and LOI.

On the other hand, the 28-day compressive strength of the mortar fabricated with the evaluated cements provided a lower correlation coefficient (0.814 in Figure 1b), and the tendency line was quite different than the equity line Estim = Experim, giving the equation Estimated $f^{28}{}_C$ = 2.1783 (Experim $f^{28}{}_C)^{0.773}$. This might be due to other chemical processes involved during paste hardening, not only related to clinker but also other SCMs, which are not provided by the manufacturers. Even so, the correlation coefficient is quite high, and this empirical equation might be used to have a rough idea of the $f^{28}{}_C$ of the mortar fabricated with a particular cement having only the information of the powder's ρ, F, and LOI.

4. Conclusions

With the results obtained with cement pastes manufactured with BPCs, the following conclusions were obtained:

The reduction of clinker content in Portland cement significantly affects the performance of cement pastes. Therefore, there is a need to estimate at least ρ, F, and LOI of these cements to indirectly estimate the ILSF content. While it would be preferable to know the clinker content (clinker factor), this is not currently mandatory in the commercialization of cements in Mexico and other Latin American countries. Knowing the cement's ρ, F, and LOI will help propose measures to counteract possible impacts on cement-based materials using these modern formulations of BPCs.

An excellent correlation was observed between ρ, F, and LOI when only ILSF was used as an addition. If another component is added to BPCs, this correlation should be verified and compared with the one obtained for ILSF. The density values of the evaluated cements varied between 2.89 and 3.15 g/cm^3, and the LOI values ranged from 2% to 18%, demonstrating that modern BPCs are extremely different from each other, even if they belong to the same strength class.

It was observed that the amount of water necessary for BPCs to achieve normal consistency increases as LOI increases. This apparently occurs because the ILSF also absorbs part of the mixing water. Since ILSF does not react with water to form hydration products that bind the aggregate together, water in excess evaporates, forming voids or pores that may be interconnected, increasing the capillarity absorption of the hardened paste. The values of the water-to-cement ratio (w/c) obtained varied between 0.25 and 0.33, suggesting that the evaluated BPCs vary significantly in their clinker and addition contents, raising doubts about the performance of the concretes where these BPCs will be used, which should also vary significantly.

The initial and final setting times (ST_I and ST_F, respectively) of BPCs evaluated in this research also showed highly varying values, and an excellent correlation was observed between ST_I/ST_F and LOI when only ILSF is used as an addition to replace the clinker. If BPC has another addition apart from ILSF, the setting times obtained were 25%–35% slower in the hardening process.

A good correlation was found between the L* coordinate obtained through a colorimetry test from the evaluated hardened cement paste specimens and the LOI parameter of the cement powder, indicating that it could be a quality control test that would help determine the ILSF content in cement.

The compressive strength at 28 days (f^{28}_C) of mortars made with the evaluated BPCs also showed notable differences depending on the addition content, mainly the ILSF content. Still, most tested cement strength classes reached the specified strength regardless of additions used and clinker content in the BPC evaluated.

With the obtained experimental data (ρ, F, and LOI), four empirical correlations were obtained to forecast the amount of water to get a normal paste consistency, setting times (initial and final), and mortar 28-day compressive strength (f^{28}_C). Three of the obtained correlation equations (for w/c ratio, ST_I, and ST_F) presented an $R^2 > 0.93$, which means that a good approximation is possible if cement powder properties (ρ, F, and LOI) are known. Only the estimates for f^{28}_C from cement powder's properties gave an empirical equation with $R^2 = 0.814$, quite far from the equality line between experimental and estimated. Still, the R^2 was quite high and may help to forecast an approximated value before the actual fabrication of the mortar to obtain the cement's f^{28}_C.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Author contributions

AATA: principal investigator, data collection, supervision, data analysis, manuscript generation, manuscript generation/editing; RAMP: specimen fabrication, data collection, data analysis, manuscript generation; CAP: specimen fabrication, data collection, data analysis; ESHS: supervision, data analysis, manuscript editing; SRR: specimen fabrication, data collection, data analysis, manuscript preparation.

Conflict of interest

The authors declare no conflict of interest.

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