

Research Article

Self-compacting mortars with mineral additions: perlite and limestone filler

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Verónica Fernanda Artigas^{*}¹, María Josefina Positieri², María Virginia Quintana³, Ángel Oshiro⁴, Franco Rodrigo Cortez⁵

- ¹ INIQUI-CONICET, Facultad de Ingeniería, Universidad Nacional de Salta, Salta (Argentina), veronicaartigas6@gmail.com
- ² CINTEMAC, Universidad Tecnológica Nacional Facultad Regional Córdoba, Córdoba (Argentina), mpositieri@gmail.com
- ³ INIQUI-CONICET, Facultad de Ingeniería, Universidad Nacional de Salta, Salta (Argentina), quintanamvirginia@gmail.com
- ⁴ CINTEMAC, Universidad Tecnológica Nacional Facultad Regional Córdoba, Córdoba (Argentina), oshiroangel@gmail.com
- ⁵ Facultad de Ingeniería, Universidad Nacional de Salta, Salta (Argentina), franco6cortez@gmail.com *Correspondence: veronicaartigas6@gmail.com

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Abstract: with the increasing infrastructure and the need for eco-sustainable alternatives, the concrete industry has motivated to look for supplementary cementitious materials especially those generated from waste. The advantages of self-compacting concrete (SCC) make it useful in the worldwide; therefore, it is essential to study additions that can be incorporated into it, to improve its properties. Limestone powder (LP) and local waste perlite fines (PF) from Salta (Argentina) are proposed as mineral addition in self-compacting mortars (SCM). PF has a high content of SiO₂ and Al₂O₃ which can contribute to increasing the strength of the concrete due to the pozzolanic reaction. Physicochemical characterization of this waste is studied and the behavior of SCM mixtures with different proportions of LP and PF are also analyzed. Sets of specimens have been performed in order to test their rheological characteristics in the fresh state and their mechanical resistance in the hardened state. The results indicate that the combination of LP and PF has a beneficial influence on the fresh state, granting an adequate fluidity and viscosity of the mixture; on the hardened state, compressive strengths similar to the standard are achieved at 90 days in mortars with up to 20% cement replacement by additions.

Keywords: mortars, self-compacting, perlite, limestone filler.

1. Introduction

Growing demand for cement in recent years has led to an increase in its production, observing a daily upward trend. World cement production is more than 4 million metric tons (Nidheesh & Suresh Kumar, 2019). The principal negative effect of the cement industry is that it generates up to 7% of the total world CO_2 emissions and consumes large amounts of energy and resources.

The Romans used mineral additions for the construction of many buildings, some of which still stand today demonstrating their strength and durability. The incorporation of mineral additions in cement and concrete industries is a topic widely treated in several publications. Depending on the type and quantity of the additions, the result can be very advantageous, e.g. a reduction in Portland cement consumption, improves its workability, giving it lower permeability, greater durability, or greater strength, among others (Erdem, Meral, Tokyay, & Erdogan, 2007). The different mineral additions in ternary and quaternary binders can have an important synergistic effect on fresh and hardened concrete properties.

Additions play a fundamental role in SCC, they contribute to reaching the necessary viscosity so the mixture can flow without presenting segregation. SCC is a concrete capable of flowing inside the formworks, passing through the reinforcing bars, and completely filling the mold, achieving compaction only by the action of its weight without presenting disintegration or exudation. SCC multiple advantages make it used worldwide, therefore it is essential to study additions that can be incorporated into it, to improve some of its properties or contribute to sustainability through the reduction of the cement content in the mix. Tests on SCM are very important to select the materials to be used and optimize their proportions in the mixtures. For this reason, SCM elaboration with different combinations of additions is projected as a preview step to avoid numerous tests at the concrete level.

Pozzolanic or cementitious materials, such as Natural Pozzolans (NP) and industrial by-products, have been used in construction, for a long time. There are two ways to use them, as an additive to clinker in cement production or directly as mineral addition in concrete production. The possible amount of pozzolans incorporated would vary according to the specific needs and the characteristics of the material (Turanli, Uzal, & Bektas, 2005). The abundance of NP and the variability in their chemical and physical composition make it necessary to develop an appropriate strategy for its use in the concrete production industry (Nicoara, et al., 2020). Pozzolans are silicate or alumino-silicate materials usually of volcanic origin.

There are many benefits of using NP in cements and concretes, mainly, a higher later age compressive strength, increased concrete durability, a lower heat of hydration and a reduced bleeding (De Bellie, Soutsos, & Gruyaert, 2018). In addition, it should be noted that the use of large quantities of this material is of vital importance for the sustainability of the cement and concrete industry, not only concerning to the energy efficiency and environment but also to the durability and economy of concrete structures (Mehta, 1998). Recently, multiple studies on NP use in SCC, have been conducted including Ghafoori, Sharbaf, & Najimi (2016), Ghasemi, Rasekh, Berenjian, & AzariJafari, (2019), Menadi & Kenai (2018). Successful results in the addition of NP in SCC will increase its use, reduce cement consumption and consequently protect the environment. The many regions with volcanic activity contain millions of tons of NP (Adekunlea, Ahmada, Maslehuddinb, & Al-Gahtani, 2015).

Perlite is a glassy volcanic rock; its chemical composition indicates that approximately 70-75% is SiO₂ and 12-18% is Al₂O₃. Its outstanding property is to expand when subjected to the action of heat, increasing its volume up to twenty times. Expanded, it is used mainly in the construction industry as thermal and acoustic insulation and also in horticultural and industrial applications. Because of its vitreous structure and high silica and alumina content perlite is considered a pozzolan (Erdem et al., 2007). However, there are a limited number of technical documents about this material pozzolanic properties (Demirboga, Örüng, & Gül, 2001; Erdem E. , 1997; Erdem et al., 2007; Karein, Joshaghani, Ramezanianpour, Isapour, & Karakouzian, 2018; Urhan, 1987; Yu, Ou, & Lee, 2003).

World reserves of perlite total 6700 million tons, approximately (De Bellie et al., 2018; Erdem et al., 2007). In Argentina, the most important perlite deposits are located in the vicinity of San Antonio de los Cobres town, in the northwestern of Salta province. They are distributed in the Quirón, Rupasca and La Ramada gorges (Gonzalvez, Herrmann, & Zappettini, 2004). The perlite rock is mined by opencast methods and then pulverized and expanded (Berge, 2009). During the mechanical process, fine material waste is generated which is deposited in the open air (Figure 1). Although this is an inert waste, it can affect the health of the inhabitants because the strong winds in the area below the dusty material away. PF used in this investigation is an example of these wastes that could be reused in the concrete elaboration and, specifically, in SCC.

On the other hand, LP is a by-product generated through the extraction process of carbonate rocks; hence, its main component is calcium carbonate, $CaCO_3$ (Adekunlea et al., 2015). This material does not possess pozzolanic properties (Bosiljkov,

2003; Liu & Yan, 2010), but has well-known effects as a partial replacement of Portland cement. The addition of LP to clinker modifies the granulometry of the mixture as it completes the granulometric curve of the cement, does not increase the water demand, improves its packing, and blocks the capillary pores. During the hydration, C_3A forms carboaluminates, which prevent the transformation of ettringite-monosulfoaluminate (Bonavetti, Rahhal, & Irassar, 2001). LP accelerates the hydration of clinker particles since it generates nucleation sites at early ages (Bonavetti V., 1998; Soroka & Stern, 1977).

Consequently, it is responsible for the improvement of initial resistance, but does not produce calcium-silicate-hydrate (C-S-H) gel. LP incorporation generates dilution effect, which consists of the reduction of the amount of cementitious material and causes the consequently effective w / c ratio increase and the strength decrease. In terms of durability, the nucleation effect of LP particles refines the pore structure of concretes (Gupta, Mohapatra, & Bansal, 2020), but problems with respect to Portland limestone cements, above all susceptibility to sulfate attack (especially, taumasite formation) and chloride ion diffusion, have been reported however, they depend on the addition level (Hartshorn, Sharp, & Swamy, 1999, 2001).



Figure 1. Fine perlite waste deposit. Source: self-elaboration.

2. Description of the problem

Over the last few years, issues related to protecting the environment and reducing production costs have been key for different industries. Cement and concrete industries are not exempted from this; they seek to promote the use of supplementary, natural, residual materials or industrial by-products to reduce energy consumption during production (Raggiotti, Positieri, Locati, Murra, & Marfil, 2015). Different industries generate wastes that are deposited directly into the environment and cause problems. An effective method to mitigate the negative effects of these wastes and contribute to sustainability is to reuse them as additions or to manufacture new products, thus natural resources are more efficiently employed, and deposits are minimized (Shirule, Rahman, & Gupta, 2012). A way to reduce local consumption of clinker, minimize CO₂ emissions and consequently save energy may be achieved by replacing part of the cement with the addition of LP and FP and, in the case of FP, using an industrial waste that currently pollutes the environment.

Therefore, in this work a limestone filler and a perlite fine waste from the mining industry from the region of La Puna in Salta (Argentina) are proposed as mineral additions in SCM; the high content of silica and alumina in perlite makes its pozzolanic reaction with Ca(OH)₂ feasible. The behavior of different SCM mixtures with proportions up to 30% LF and up to 30% PF for cement individually and up to 50% combined are also analyzed. Sets of specimens have been performed in order to test their rheological characteristics in fresh state and their mechanical resistance in hardened state. Workability is determined by using V-funnel test and slump-flow test. Prismatic specimens were tested for flexion and compression at 1, 3, 7, 28 and 90 days.

3. Methodology

This research was divided in two parts. First, FP mineral addition was physically and chemically characterized to determine its activity and application as a pozzolan. In a second stage, different proportions of FP (10, 20, and 30%) combined with LP were incorporated to design different mortar mixtures. The fluidity and viscosity of the mixture were measured by slump flow

test and the V-funnel for mortars, following the ASTM C1611/C1611M (2018) standard and the specifications and guidelines for self-compacting concrete EFNARC (2002). The compressive strength was determined at 1, 3 7, 28, and 90 days, according to the IRAM 1622 (2002) standard.

3.1. Characterization of the pozzolanic material

The mineral addition (FP) has a density of 2.23 g/cm³ (determined by the pycnometer method) and a BET specific surface area of 5.28 m²/g. The grading distribution of the sample by laser diffraction performed on a HORIBA LA-950 particle size analyzer can be observed in Figure 2 (left); approximately 90% of the particles are smaller than 75 μ m. The chemical composition of perlite material is defined by SiO₂ (74.14%), Al₂O₃ (13.49%), Fe₂O₃ (0.66%), Na₂O (4.14%), K₂O (4.24%), CaO (0.59%).

The material was also analyzed by X-ray diffraction (XRD), was carried out with a Philips PW1800/10 diffractometer, working at 40 kV and 30 mA. X'Pert High-Score (PANalytical) software was used to quantify the mass fractions of the mineral phases identified by XRD. The diffractogram from the perlite sample (Figure 2, right) shows characteristic peaks of silica minerals (such as quartz) that have their main peaks between 20-22° and 26-28°. A curved design for the region between 40-90° is also observed at the bottom of the diffractogram; this could be due to the lack of a defined mineral structure in the analyzed material.



Figure 2. (left) FP grading. (right) FP diffraction pattern.

3.2. Materials

Cement: a class 40 resistant pozzolanic Portland cement (PPC 40) was used, which complies with the IRAM 50000 (2014) standard. The PPC includes pozzolan, in its composition, up to 35% according to that standard. PPC 40 has a density of 3.10 g/cm³. There is no record of experimentation on mortars with CPP and PF; this cement was used because of its local commercial availability, considering a possible transfer to the local construction industry.

Aggregates: the fine aggregate used was natural siliceous sand, it has continuous granulometry (maximum size 4.75 mm) and is within the limit curves, in accordance with standard IRAM 1505 (2005) and IRAM 1627 (1997). The aggregate has a density of 2.65 g/cm³, absorption of 0.91%, and a fineness modulus of 2.23.

Additive: a superplasticizer additive based on polycarboxylate was used. The additive has a solid content of 35%. The dose used was within the limits recommended by the manufacturer, it was considered as a percentage of the weight of the cementitious material. The additive complies with IRAM 1663 (2002) and the requirements of ASTM C494/C494M (2013) for type A, water-reducing additives.

Addition: FP waste from a quarry in San Antonio de los Cobres, Salta Province, Argentina was used as a potential pozzolanic material. This addition contains mainly silica and alumina, 90% of its particles are smaller than 75 microns. FP has a density of 2.23 g/cm³. LP for commercial use was used as a filler material, its composition is defined by CaCO₃ (93%±3), Ca⁺² (36.8%±1.2), Mg⁺²(1.0%), and Fe (0.5%); 90% of its particles are smaller than 300 microns. LP has a density of 2.60 g/cm³.

3.3. Methodology and equipment for tests in fresh and hardened state

It is relevant to know the fluidity and viscosity of the mortars as they condition the filling capacity and resistance to segregation of the concrete. For this purpose, flow cone and V-Funnel tests were carried out on different mortar. To evaluate if the properties in fresh state were maintained the test times considered were 6 and 45. Figure 3 shows the equipment used in the V-funnel test and its dimensions. The test belongs to the orifice type and consists of filling the funnel with mortar and measuring the time it takes to drain through the lower opening of the funnel. An increase in time through the V-Funnel indicates a decrease in the viscosity of the mixture. Figure 4 shows the cone used in the runoff test and its dimensions. As a first step, the cone is filled with the mortar mixture; then, the cone is lifted with a smooth movement, and two perpendicular readings are taken of the diameter reached by the slump-flow. The final diameter is the average of both values.



Figure 3. (a) V-funnel. Source: Self-elaboration; (b) V-funnel dimensions. Source: Adapted from EFNARC (2002).



Fifteen prismatic specimens for each type of mortar were molded to evaluate the mechanical properties. The specimen dimensions were 40 x 40 x 160 mm. They were kept in a curing tank until the corresponding test age, three samples of each mortar were tested per age. Each specimen was subjected to a flexural test, which generates two halves of the sample. Then, each half was tested in compression, determining its breaking load. The final compressive strength of each specimen is the average of the two readings. Figure 5 shows the tests devices.



Figure 5. (a) flexural strength testing device; (b) compressive strength testing device.

3.4. Design of mortars samples with the incorporation of perlite fines and limestone filler

A mortar was designed of reference and fourteen mixtures were made in which the amount of cement substituted (10, 20, 30, 40 and, 50% by weight) was changed to LP and FP. The cementitious material-water ratio was kept constant at 0.4. The amount of additive used in all the mixtures was the one necessary to achieve a 30 cm slump flow in the control sample (without substituted cement), being 0.85% of the cementitious material in weight. These dosages are shown in Table 1.

Mixture	Cement (kg)	Water (kg)	LP (kg)	FP (kg)	Sand (kg)
C100	700		0	0	1288
C90F10	630		70	0	1278
C90P10	630		0	70	1266
C80F10P10	560		70	70	1257
C80F20	560		140	0	1269
C80P20	560		0	140	1245
C70F30	490		210	0	1259
C70P30	490	280	0	210	1224
C70F10P20	490		70	140	1236
C70F20P10	490		140	70	1247
C60F20P20	420		140	140	1226
C60F30P10	420		210	70	1238
C60F10P30	420		70	210	1214
C50F30P20	350		210	140	1217
C50F20P30	350		140	210	1205

4. **Results and discussion**

Figure 6 shows slump-flow test and V-funnel results at 6 and 45 minutes after the mortar preparation was started, the effects of the additions in the fresh state are appreciated. The incorporation of LP into the mixture increases the values of the slump-flow diameter and decreases the times of passage through the funnel. It indicates that the LP filling and dilution effect improves flowability and lowers the viscosity. FP produces the opposite results, in these mixtures the differences between the results obtained at 6 and 45 minutes are greater, with an increase in the loss of workability over time. Fresh state tests could not be performed in mortars with 30% FP at 45 minutes, since the mixture did not have the necessary flowability.



Figure 6. Slump-flow test and V-funnel test results. Source: Self-elaboration.

Figure 7 shows iso-response curves of fresh state test results at 6 minutes for mortars in study. The results of slump-flow tests suggest that in the mortars with a higher amount of LP, there is a further increase in slump-flow values compared with the control and those containing FP. This effect was also observed by Uysal & Yilmaz (2011) and might occur by the increased surface area of the FP particles ($5.28 \text{ m}^2/\text{g}$) concerning the cement (around $1 \text{ m}^2/\text{g}$) which increases the water demand. The water content was kept constant for all of the mortars elaborated in this research. So, LP needs for water is less than FP which has provided more slump-flow. In mortars with a combination of both additions, the mentioned effects are counteracted. For example, C70F10 achieves a slightly higher slump-flow than the control mortar, while C70P10 only achieves 72% of that value; however, the slump-flow of the C70F10P20 and C70F20P10 mixes are approximately 96% of the standard.



Figure 7. (a) Iso-response curves of V-funnel test results at 6 minutes (s); (b) iso-response curves of slump flow test results at 6 minutes (cm).

Furthermore, FP has higher volume compared to the same weight of LP or Portland cement due to the lower density of FP than LP and Portland cement; therefore, the solids volume in the paste and the contact between their particles increase. SCM produced with FP requires a larger amount of mixing water for lubrication than SCM control and with LP. It is seen that the viscosity of C70P30 series containing only FP is quite higher than the other series, which was reflected in the increase of passage time values through the funnel. In this case, the effects are also balanced by the mix of the additions.

Although the results of the mixtures that only had LP reflected good behavior, it is necessary to note that they presented a

certain degree of segregation in their mass, since LP, in such low proportions as 30%, does not provide the conditions of viscosity required by this type of mortar. In addition, the mixtures with FP had higher viscosity as their content increased. This coincides with that indicated by De Bellie et al. (2018) for natural pozzolans, they tend to have a flocculant structure when incorporated into mortars, so more cohesive mixtures with higher water retention capacity are obtained. Figure 8 presents slump-flow tests conducted on the mortars. The mixture with higher amounts of LP does not present a homogeneous distribution of aggregate particles; they are concentrated in the central part of the flow that indicating slight segregation. On the other hand, the mixture with FP presents a visually more homogeneous flow without indices of segregation.



Figure 8. Slump flow tests on mixture with LP (left) and with FP (right).

Figure 9 shows the compressive strength at different ages (1, 3, 7, 28, and 90 days); each value represents the test average of three specimens. The evolution of resistance is different with the incorporation of each percentage and each addition. For mixtures with greater FP than LP content, strength gain between 7 and 28 days is lower than that of the rest. On the contrary, at higher content of FP, the mixture presents a greater late age strength increase, which confirms the pozzolanic properties of this addition. Results agree with Erdem et al. (2007) and Cobirzan, Balog, & Mosonyi (2015), who obtained a lower compressive strength than the baseline when they replaced (20% - 30%) cement with perlite from different sources.



Figure 9. Compressive strength at different ages. Source: Self-elaboration.

Figure 10 illustrates the iso-response curves of compressive strength showing the interaction effect of FP and LP for the domain studied. Strength decreases as cement replacement is greater for all ages and both additions. At 1 day this difference

in resistance is more evident, for example, the mortars with 50% cement replacement (C50F30P20 and C50F20P30) had a resistance of 40% concerning the pattern mortar. At 7 days they achieved a resistance of 44%, while at 90 days they reached 63%.

The mortar with 30% FP achieves only 62% of its final strength at 28 days, while the standard mortar has already achieved almost 100% of its strength.



Figure 10. Iso-response curves of compressive strength at different ages (MPa); (a) at 1 day; (b) at 3 days; (c) at 7 days; (d) at 28 days; (e) at 90 days.

The combination of FP and LP does not give good results from the point of view of compressive strength for cement replacements greater than 30%. At 90 days, the C80F10P10 mortar reaches a strength in the range of the pattern, while replacing 30% of the cement (C70F10P20 and C70F20P10 mortars) only reaches 81%, with 40% replacement (C60F20P20, C60F30P10, and C60F10P30) 73% of the standard strength and with 50% combined additions in the mix (C50F30P20 and C50F20P30) only 64%.

5. Conclusions

Based on the analysis of the results of this research the following conclusions can be made.

- 1. The addition of FP, concerning the pattern mortar, decreases the fluidity and increases the viscosity of the mix, thus requiring an increase in the dosage of SF admixture to maintain these conditions. In addition, it produces a greater loss of fluidity over time. In the hardened state, it decreases the compressive strength at early ages for all percentages, but at advanced ages, it achieves resistances similar to the pattern for replacements of up to 10%.
- 2. The addition of LP increases the fluidity and decreases the viscosity of the mixture concerning the pattern mortar. Mortars with LP show higher strength development at 28 days compared to FP mortars for all percentages; at 90 days both achieve similar strengths for the same percentage of replacement.
- 3. The combination of FP and LP (for all replacement percentages) is beneficial in the fresh state as they combine high flowability and moderate viscosity, reducing the segregation of the mixture. The combination of these additions as partial cement replacement in SCM is feasible from the mechanical point of view, reaching final strengths in the order of the pattern for replacements up to 20% cement (10% LP and 10% PF). For cement replacements by FP and LP over 30%, the final strengths are considerably lower than the pattern.
- 4. Although the achieved strength values are not as high as expected for an SCM, it can be said that these values meet the standards in structural projects when a moderate strength is required (in agreement with the premise that mortar mechanical properties will be reflected in the concrete on which it is based). Moreover, it should be noticed that the use of finely ground raw perlite waste in percentages of replacement up to 20% in mortars contributes to sustainability, saving energy and resources, and reducing CO₂ emissions generated during the manufacture of Portland cement clinker. Additionally, it prevents this waste material from affecting the environment.

Finally, the results obtained in SCM provide information to be transferred to the use of FP and LP in SCC.

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