Iron slag as fine aggregate replacement and nanosilica particles in self-compacting concrete mixtures

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Abstract. A study of the mechanical behavior of self-compacting concrete mixtures under sand replacement (as fine aggregate) by iron slag (residue from industrial machining) from 0.0% to 50.0% of mass, variations of water/cementitious material ratio between 0.3 and 0.5 and nano SiO₂ incorporation between 0.0% and 2.0% by mass of cementitious materials is presented. Fresh state tests of slump flow were performed, and the main rheological parameters: static yield stress and plastic viscosity were determined from a rheometer. For the hardened state, compressive strength tests were performed. The study of iron slag incorporation and water/cementitious materials ratio variation was developed based on a statistical methodology of central composite design from axial points based on a 2k factorial with central points, besides a posterior analysis of variance and Tukey’s multiple comparison tests. The optimization of these variables was developed using response surface methodology on 7 days compressive strength results, from the statistical design, besides the posterior determination of nano SiO₂ effects on the optimized proportions. Among the most relevant results regarding the presence of iron slag, an increase in the early age compressive strength was found, with the optimized mixture strength being more than 100% higher than mixtures without iron slag at 7 days of curing. Regarding the effect of nano SiO₂ addition to the optimized mixture, a detriment of the rheological parameters and a consequent reduction of the workability were the most remarkable findings. With the obtained results, iron slag proves to be a feasible sand replacement in self-compacting concrete mixtures.

1. Introduction
Nowadays, self-compacting concrete (SCC) is being widely used due to its properties and characteristics, as it does not need vibration in its placement, flows under its own weight and, properly proportioned, is capable of filling formwork without difficulties and without bleeding or segregation [1]. SCC consists of modifications such as: a greater amount of fine particles, the use of superplasticizer and/or viscosity modifying admixtures, besides the partial replacement of cement by supplementary cementitious materials (SCM). The commonly used SCM are fly ash, ground granulated blast furnace slag (GGBFS), silica fume, metakaolin, among others, and its benefits in the mixtures varies from fresh state improvements in workability and less heat of hydration to higher strengths in hardened state, while decreasing the amount of cement per cubic meter of concrete [2].

Concrete making leads to problems such as depletion of natural resources as fine aggregate, which mining generates negative impacts on river ecosystems [3]. The annual global concrete consumption amounts to roughly 25 billion tons, within which the fine aggregate constitutes a quarter of the total volume of concrete matrix, its high consumption has marked a decrease in the availability of quality fine aggregate, producing depletion and making extraction difficult, which has forced certain countries to...
impose restrictions on its extraction [4]. Recently [5,6] it has been found that iron slag (IS), a byproduct of iron works processes in industrial machining workshops, potentially improves the compressive strength of concrete when used as sand replacement, thus proposing a solution to the problem of resources depletion together with mitigation of the environmental impact of its disposal.

Traditional fresh state tests, as workability ones, do not give data with enough confidence because they are empiric and subjective [7]. So, rheology, as the science that studies the materials that flow [8], allows to determine concrete fundamental physical parameters as plastic viscosity and yield stress. The former measures a material resistance to flow, and the latter, the required stress for a material to start flowing; hence, the study of these parameters provides a scientific vision of the concrete fresh state behavior [9]. The use of synthetic materials, like nano-particles of SiO₂ (nS), as a material for concrete production is still novel [10]. Even so, several studies show improvements in the mechanical strength and durability properties of concrete and cement based materials by the use of nS [10,11].

Therefore, this document contains an analysis of several fresh and hardened state SCC properties with variations of IS and nS amounts, and water/cementitious materials (w/c) ratio. The analysis was done by means of a statistical central composite design (CCD) based on a 2⁸ factorial, with IS amount and w/c ratio as the statistical design factors, and with response surface methodology. This, in order to evaluate the incidence of sand replacement by IS and the simultaneous variation of w/c ratio. Slump flow as well as rheology tests were performed for fresh state. In hardened state, compressive strength tests at 7, 14 and 28 days were carried out. The analysis of the results from each of the tests was performed using ANOVA analysis of variance and Tukey’s multiple comparison test. Optimization from response surface analysis was carried out with the results of 7 days compressive strength in addition to the determination of the effect of nS particles incorporation on optimized results. This investigation aims to promote the use of a considerably new technology (i.e. SCC) in Colombia, which is of still limited use. Additionally, this document would encourage the use and research of new alternative materials for concrete production by researchers and the Colombian industry. For IS, an industry byproduct, this investigation would promote an alternative for its disposal and possible use as a concrete raw material.

2. Materials and methods

2.1. Materials
The materials used in this investigation were Portland cement (PC) Type III (high early strength), GGBFS, coarse aggregate (CA), fine aggregate (FA), water, IS, superplasticizer (SP) and nS; GGBFS and IS were used as cement and fine aggregate replacements, respectively. The characterization of the materials used was carried out following the parameters of the American Society for Testing and Materials (ASTM). PC and GGBFS specific gravity (SG) were 3.08 and 2.85, respectively. CA and FA were obtained from local sources, with SG of 2.80 and 2.73, respectively. SG of IS was 5.21. Fineness modulus of FA and IS was 1.53 and 3.03, respectively. CA with nominal maximum size of 12.7 mm. IS was subjected to a treatment in Los Angeles abrasion machine due to its original size and to improve its granulometry. Commercially available polycarboxylate-based superplasticizer with 43% of solid content and SG of 1.08 was used. The nS used was an aqueous dispersion (slurry) with a SG of 1.70 and solid content of 31.54%, with particles size up to 30 nm.

2.2. Statistical methodology
Central composite design, based on factorial 2⁸ experiment design, is a statistical tool to investigate the effects of the experiment factors in a response. With the use of a central point it is possible to determine the effects and interaction of the factors, and to optimize from a response surface without having to execute the full factorial design, saving experimentation time and costs [12]. This statistical methodology also offers objective decision making with confidence levels (instead of descriptive statistics) [13] and it has been proven helpful by several authors [12,14,15] in cement composites investigations when the effects and ranges of variables are unknown, like the IS in the present study.
The current work uses two factors, w/c ratio and iron slag percentage (%IS), where the base 2 represents the levels of the factorial part of the design and \( k \) is the number of factors. %IS varied from 0.00% to 50.00% and w/c ratio varied from 0.30 to 0.50 and. The limits of the factors ranges were chosen both from literature review and from field experience. The few investigations with the use of iron slag reached replacement limits up to 40% of fine aggregate mass [5,6,14,16]. The w/c ratio limits were chosen in order to fulfill EFNARC requirements for SCC [17]. Mixtures with ratios outside this range are difficult to perform and compromise its quality. In this study, the slump flow, the static and dynamic rheological parameters and compressive strength at 7, 14 and 28 days, represented the responses, and optimization was performed on 7 days compressive strength (CS). 7 days CS was chosen as the optimization target in order to test the capacity of the statistical methodology to predict at concrete early ages, which is of interest for the precast concrete industry [18] for which SCC is commonly used. The incorporation of nS was tested on the optimization result with 2% for nS limit, which was selected as it falls in the range for usual improvements in concrete due to its addition, as the review articles investigated offered [10,11]. The experimental statistical design presented nine points as combinations of the used factors (\( P_1 - P_9 \) treatments), the external points with double replica, while the central point (\( P_0 \)), was replicated 10 times for a total of 26 statistical and independent batches. Table 1 shows the nine points previously mentioned and the combination of w/c ratio and %IS. The analysis of variance (ANOVA), which finds statistical significant factors, and Tukey’s multiple comparison test, which tells whether there is statistical difference among treatments, were performed with an established significance level \( \alpha = 0.10 \).

2.3. Mixtures proportions

Mixture designs were done according to the recommendations proposed by the American Concrete Institute (ACI) in the ACI 211.1-91 [19] and with some modifications from EFNARC [17] to achieve self-compactability conditions. GGBFS was used as constant cement mass replacement by 20% and the fine aggregate content was kept 30% higher (by mass) than the coarse aggregate in all the mixtures, this because preview slump-flow tests carried out with this relationship exhibited the best behavior. Table 1 provides details for w/c ratio and IS replacement percentages (w/c ratio | %IS) in accordance with the statistical treatments and materials amount per mixture.

| Mix description | PC  | CA  | FA  | GGBFS | IS  | Water | SP  |
|-----------------|-----|-----|-----|--------|-----|-------|-----|
| \( P_1 \) (0.33 | 42.68) | 437.29 | 721.71 | 537.92 | 109.33 | 400.50 | 190.25 | 3.82 |
| \( P_2 \) (0.47 | 7.32) | 305.92 | 791.25 | 953.75 | 76.50 | 75.00 | 194.17 | 2.67 |
| \( P_3 \) (0.40 | 25.0) | 360.00 | 762.92 | 742.92 | 90.00 | 247.50 | 191.67 | 3.14 |
| \( P_4 \) (0.40 | 0.00) | 360.00 | 765.37 | 995.33 | 90.00 | 0.00 | 194.00 | 3.14 |
| \( P_5 \) (0.40 | 50.0) | 360.00 | 761.25 | 482.54 | 92.33 | 493.79 | 190.87 | 3.14 |
| \( P_6 \) (0.50 | 25.0) | 288.00 | 798.58 | 779.17 | 72.00 | 258.33 | 191.67 | 1.67 |
| \( P_7 \) (0.33 | 7.32) | 437.25 | 725.92 | 874.79 | 109.33 | 69.08 | 192.38 | 3.81 |
| \( P_8 \) (0.30 | 25.0) | 480.00 | 702.62 | 684.92 | 120.00 | 228.29 | 190.71 | 4.18 |
| \( P_9 \) (0.47 | 42.68) | 305.83 | 787.75 | 587.08 | 76.25 | 437.08 | 192.29 | 2.22 |

2.4. Performed tests

2.4.1. Fresh state tests: The flowability of the concrete was evaluated through the slump flow test (SFT) using the Abrams cone according to the recommendations given by the European Federation for Specialist Construction Chemicals and Concrete Systems (EFNARC) [17].

2.4.2. Rheology: For the rheology tests, the ICAR RHM-3000 rheometer from the International Center for Aggregate Research (ICAR) was used. This device, which consists in a rotatable vane and a concrete recipient, allowed to determine the static yield stress (SYS) and plastic viscosity (PV) of the concrete.
2.4.3. Hardened state tests: Compressive strength (CS) tests were carried out on \( \Phi 100 \times 200 \) mm cylindrical specimens at 7, 14 and 28 days of curing. Three specimens were tested for each batch and for each day of curing. The specimens were tested for CS according to ASTM C39 [20] procedure.

3. Results

ANOVA analyses were conducted on the tested properties of the concrete, and the experimental parameters, whose significance was the aim of the analyses were: the linear effects of the variables (%IS and w/c), their squared effects (%IS\(^2\) and w/c\(^2\)) and their interaction effect (%IS⋅w/c). Table 2 shows the significant variables for each of the performed tests, lack-of-fit significance and \( R^2 \) fitting values. The terms presented in Table 2 proved to be statistically significant (p-value < \( \alpha \)). Lack-of-fit, which is a mathematical assessment of the adjustment of the model order to the experimental data, proved not to be statistically significant (p-value > \( \alpha \)) for the studied concrete properties, with null hypothesis being that the chosen variables were enough to explain the behavior of the data. Additionally, it was carefully guaranteed that the data fulfilled the ANOVA assumptions of normality, independency and equality of variances [13], indicative of the mathematical validity of the experiment.

| Table 2. Summary of ANOVA results. |
|-----------------------------------|
| Studied properties | Significant variables | Lack-of-fit (p-value) | \( R^2 \) (%) |
|---------------------|-----------------------|----------------------|--------------|
| Slump flow | w/c, %IS, w/c\(^2\) | 0.73 | 65.51 |
| Static yield stress | w/c, %IS | 0.84 | 30.47 |
| Plastic viscosity | w/c, %IS\(^2\) | 0.64 | 43.27 |
| 7 days CS | w/c, %IS, w/c⋅%IS | 0.79 | 79.21 |
| 14 days CS | w/c, %IS | 0.85 | 76.87 |
| 28 days CS | w/c, %IS | 0.85 | 77.79 |

With the purpose of determining the influence of factors levels variations on the responses when keeping constant the other factor levels, Tukey’s multiple comparison test was performed. Table 3 shows the test results when keeping constant the w/c ratios and Table 4 shows the results when keeping %IS constant. Mean values that do not share a letter (a, b or c) are statistically different.

| Table 3. Tukey's multiple comparison results for constant w/c ratio. |
|---------------------------------------------------------------|
| w/c ratio | 0.33 | 0.40 | 0.47 |
| %IS     | 7.32 | 42.68 | 7.32 | 42.68 |
| 7 days CS (MPa) | 36.83 (a) | 55.69 (b) | 29.23 (a) | 36.83 (a) | 39.60 (a) | 26.72 (a) | 27.07 (a) |
| 14 days CS (MPa) | 57.72 (a) | 64.46 (a) | 39.03 (a) | 47.65 (a) | 52.82 (a) | 33.79 (a) | 35.13 (a) |
| 28 days CS (MPa) | 65.82 (a) | 76.08 (a) | 45.96 (a) | 53.97 (a) | 61.28 (a) | 38.83 (a) | 42.20 (a) |
| SFT (mm) | 713.30 (a) | 741.70 (a) | 661.70 (a) | 697.70 (a) | 713.30 (a) | 660.00 (a) | 666.70 (a) |
| SYS (Pa) | 197.30 (a) | 325.40 (a) | 298.00 (a) | 328.40 (a) | 520.00 (a) | 305.50 (a) | 484.00 (a) |
| PV (Pa⋅s) | 58.50 (a) | 88.20 (a) | 65.70 (a) | 41.27 (a) | 62.60 (a) | 30.55 (a) | 26.20 (a) |

| Table 4. Tukey's multiple comparison results for constant %IS. |
|---------------------------------------------------------------|
| w/c ratio | 0.33 | 0.47 | 0.33 | 0.47 |
| %IS | 7.32 | 25.00 | 42.68 | 42.68 |
| 7 days CS (MPa) | 36.83 (a) | 26.72 (b) | 54.76 (a) | 36.83 (b) | 22.66 (c) | 26.72 (a) | 27.07 (b) |
| 14 days CS (MPa) | 57.72 (a) | 33.79 (b) | 67.30 (a) | 47.65 (a) | 33.36 (b) | 64.46 (a) | 35.13 (b) |
| 28 days CS (MPa) | 65.82 (a) | 38.83 (b) | 77.40 (a) | 53.97 (b) | 40.10 (b) | 76.08 (a) | 42.20 (b) |
| SFT (mm) | 713.30 (a) | 666.70 (a) | 748.30 (a) | 669.70 (a) | 713.30 (a) | 666.70 (a) | 666.70 (a) |
| SYS (Pa) | 197.30 (a) | 305.50 (a) | 420.00 (a) | 298.00 (a) | 152.00 (a) | 325.40 (a) | 484.00 (a) |
| PV (Pa⋅s) | 58.50 (a) | 30.55 (a) | 64.40 (a) | 41.27 (a) | 30.50 (a) | 88.20 (a) | 26.20 (a) |

3.1. Fresh state analysis

Considering the results presented in Table 3 and Table 4, the variations in the static yield stress and the plastic viscosity were not statistically different for any of the variations of the factors, i.e., w/c or %IS,
levels. These results also agree with the low $R^2$ found in the ANOVA analysis for the SYS and the PV. Since the change in w/c ratio and %IS does not statistically affect the SYS and the PV for the analysed levels of the variables, the variation in the mean results could also be attributed to the change in SP dosage. It is to be remarked that SP dosage was modified in order to fulfill the EFNARC [17] fresh state limits for slump flow (650-800 mm), therefore, SP amount was not a controlled factor in this work.

SFT results presented in Table 4 indicate that for high %IS (25.00 and 42.68) there is statistical difference among w/c ratio variations. For %IS = 25.00 there was statistical difference between w/c ratio of 0.30 vs. 0.40 and 0.50, without statistical difference among the last two. For %IS = 42.68, there was statistical difference between w/c ratio of 0.33 vs. 0.47. This behavior could be explained by the fact that SP dosage was higher for low w/c ratios (0.30 and 0.33) because low w/c ratios decrease the fluidity of the mixtures, thus, SP was required to produce greater spread diameters in order to comply with EFNARC limits [17]. For low w/c ratios (0.30 and 0.33) the SCC have less water to disperse the cement particles, so SP increments compensate and produces even greater spread diameters than those produced by the combination of higher w/c ratios (0.40, 0.47 and 0.50) and lower SP dosage [21].

3.2. Compressive strength analysis
Table 2 shows that for the three tested ages the two variables were statistically significant, but interaction effect was significant only on 7 days CS, which shows a dependency of IS contribution to CS due to microstructure hydration progress [21]. Additionally, from the results presented in Table 3, maintaining constant the w/c ratios, the only statistical difference is observed for 7 days compressive strength with w/c = 0.33, among %IS = 42.68 and 7.30. This result could indicate a contribution to a greater compressive strength due to a higher amount of IS in the mixture. For the other concrete ages and w/c ratios, a modification in the %IS did not statistically contribute to the compressive strength development. Even though, this statistical analysis indicates the viability to use IS as partial sand replacement without compromising the strength, as it does not weaken it. This goes in accordance with results from Table 4, in which, for 7 days CS every variation in the w/c ratio produced a statistically different CS for each of the %IS. At 14 and 28 days, and for %IS = 25.00, Tukey’s test showed statistical difference between 0.30 vs 0.40 and 0.50, with the last two not being statistically different. This could be explained considering the fact that concrete compressive strength depends on the proximity of the cement particles in the hardened matrix instead of in the amount of hydration products formed [21], hence, for higher w/c ratios as the case for w/c = 0.40 and 0.50, the additional water would contribute to diminish the compressive strength as the cement particles get more dispersed. In practical terms, for 25.00% of IS, the results found, indicate the possibility of producing SCC without statistical difference in strength with the use of higher w/c ratio (0.50), which results in less expensive SCC production.

3.3. Optimization
Using the response surface methodology, an optimization of the 7 days CS, was carried out, generating the model presented in Equation (1).

$$\text{CS [MPa]} = 48.9 - 49.1 \ast \frac{W}{c} \text{ratio} + 1.863 \ast \%IS - 3.55 \ast \frac{W}{c} \text{ratio} \ast \%IS$$

(1)

The optimization of the previous equation resulted: w/c ratio = 0.30 and %IS = 50.00 as the combination of the factors to generate the maximum value for 7 days CS. Figure 1 shows the generated surface model. Additionally, in Table 5 are shown the results of the optimized proportions and the effect of nano SiO$_2$ incorporation up to 2% of cementitious materials mass in the optimized mixture. From the response surface graph (Figure 1) can be seen a tendency for lower compressive strength values as the w/c ratio gets closer to 0.50. Also, for w/c ratio of 0.30 the slope of the curve becomes steeper as the %IS increases, reaching the highest CS value when %IS = 50.00, which shows a trend for increments of the compressive strength with the evolution of IS incorporation. Since the maximum CS value of the regression model from Equation (1) (74.07 MPa) is in the statistical limits of the experiment, it could indicate the possibility to reach higher strengths with higher amounts of IS, that is, outside the limits of
the present investigation. This effect of the IS incorporation relates to the few previous experimental investigations [5,6], which found CS growth with sand replacement up to 40% by IS.

The predicted 7 days CS (from the surface model) was 74.07 MPa and the experimentally obtained strength was 79.23 MPa, as shown in Table 5, with a difference of 6.51%. According to this result, the response surface model has a good accuracy predicting the value of early age compressive strength. A diminish in the compressive strength of the mixtures with nS is also seen in Table 5. This could be an indication of too low amounts of nS for it to manifest significant improvements, as previously reported [22]. As per the fresh state of the mixtures, slump flow values diminished and rheological parameters values increased with the incorporation of nS, this behavior, previously found by other authors [12] might be due to the high reactivity and specific area of the nS, which generates attraction to water molecules, producing a decrease in the fluidity of the mixtures. Even though workability is affected, slump flow values fall inside EFNARC [17] limits (650-800 mm), meaning that, as per fresh state behavior of the mixtures, nS are suitable to be used in SCC with IS as partial sand replacement.

![Figure 1. Response surface for 7 days compressive strength.](image)

| Table 5. Optimization and nS incorporation results. |
|---------------------------------------------------|
| **Performed tests** | **0% nS** | **1% nS** | **2% nS** |
| Slump flow (mm)     | 780.00    | 716.60    | 683.30    |
| Static yield stress (Pa) | 144.80    | 762.40    | 830.70    |
| Plastic viscosity (Pa·s) | 138.90    | 159.60    | 160.40    |
| 7 days CS (MPa)     | 79.23     | 68.99     | 69.56     |

4. Conclusions
The study of the influence of iron slag as fine aggregate replacement, nano SiO₂ addition and water/cementitious material ratio variation was performed on self-compacting concrete mixtures. Iron slag proved to be a feasible sand replacement as fine aggregate, since it did not show any statistical difference in compressive strength test at 14 and 28 days of curing with replacement percentages from 0.00 to 50.00% by mass. In addition, for 7 days of curing and low water/cementitious materials ratio, iron slag increments evidenced to improve compressive strength of concrete. Nano SiO₂ incorporation did not have a remarkable effect on the early age compressive strength of iron slag self-compacting concrete mixtures, but its fresh state was affected with a decrease in its workability, as rheology analyses showed. Further work is to be developed to determine the effect of nano SiO₂ in later ages of curing of self-compacting concrete mixtures with iron slag, and with greater range of iron slag replacement percentage, since the results of the optimization were in the experimental limits of this work.
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