Investigation of the Effects of Calcite and Blast Furnace Slag in Self-Consolidating Concrete

Murat GÖKÇE¹, Burçin Şenol ŞEKER²,*, İlhami DEMİR³, Osman ŞİMŞEK⁴

¹Assistant Professor, Amasya University, Architectural Faculty
²Associate Professor, Amasya University, Architectural Faculty
³Professor, Amasya University, Architectural Faculty
⁴Technology Faculty, Civil Engineering Department, Gazi University, Turkey

*Corresponding author: Burçin Şenol ŞEKER, Associate Professor, Amasya University, Architectural Faculty, E-mail: senol.seker@amasya.edu.tr

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Abstract

A rapid development of admixture technology has accelerated the development of concrete technology. The self-consolidating concretes (SCC) were put into practice by developing polycarboxylate-based chemical admixtures. This interaction of polycarboxylate based admixtures with powder materials affects the fresh and hardened characteristics of the concrete at meso and macro levels. In the present study, the interaction of powder material paste with admixtures in the SCC was investigated at nano-micro and meso levels. In addition, a number of concrete tests were performed at a macro level. Of blast furnace slag (BFS), 10%, 20%, 30%, and 40% were substituted with cement for reference samples, while 10%, 15%, 20%, and 25% of calcite were added to the cement. Scanning electron microscope (SEM) analysis of hardened powder material paste was performed at a micro level. Rheological features and mini flow values of powder material paste were identified at meso levels. For the concretes prepared at macro levels, consistency of fresh state, the stability of consistency, specific bulk density, L-box test, a compressive strength of hardened concrete samples on days 1, 3, 7, 28, and 90 were found. With the increase in the calcite admixture, the meso level plastic viscosity results showed an increase, and the passing capacity through the macro level reinforcements was determined to have increased, as well.

It was determined that the compressive strength test of the BFS at the macro size was a positive contribution of BFS to the compressive strength for 28 and 90 days samples. As new C-S-H gels were formed as a result of the reaction of BFS with calcium hydroxides, the amount of calcium hydroxide was decreased in SEM analysis of hardened paste.
Introduction

Nowadays, while making concrete designs, applicable concrete becomes important. The process between the production and placement of the designed concretes to the mold, as well as their service life after casting, are considered. For this purpose, the selection of suitable chemical additives is also important for the manufacture of applicable concrete. When powdered materials that form concrete are mixed with water and chemical additives, chemical reactions start to occur. This interaction, beginning in the nano and microstructure, changes the meso and macro level performance of the concrete by affecting its fresh and hardened properties. In order to modify and improve the properties of the concrete at a macro level, it is necessary to better analyze the interaction at the nanoscale level.

Self-Consolidating Concrete

New generation polycarboxylate-based additives first appeared in Japan in the late 1980s to prevent loss of consistency, improve processability and solve the vibration problem in concrete. The self-compacting concrete produced using polycarboxylate based admixtures is a high-performance concrete with its own weight, which can be spread homogeneously to the desired cross-section, with full filling in the vibration-free section and with high strength properties [1].

SCC should contain more paste than conventional concrete. This makes it possible to reduce the friction between the aggregates and allow the SCC to flow and settle with its own weight. In order to increase the volume of the powder, increasing the amount of cement not only increases the cost of concrete but can also initiate problems with high shrinkage and hydration heat formation. Therefore, in order to increase the powder volume, it may be more suitable to increase the amount of fine aggregate (0-4 mm) present in the concrete structure and consequently to produce SCC with the appropriate fine material (0.125 mm sieve). Research on the rheology of both the powder paste and the concrete is of paramount importance for the improvement of SCC mixture calculations. In particular, the research and understanding of the rheological properties of the powder material pulp (0.125 mm sieve under-limestone powder, cement, water, and polycarboxylate based superplasticizer mixture) in SCC design will be very useful for achieving the targeted performance characteristics in SCC production [2-5].

Working Mechanism of Fluidizer Chemical Additives

The plasticizer chemical additives reduce the need for water for the processing and application of concrete and maintain its consistency for a certain period of time. The dispersion mechanism of these additives depends on two different types of impulse forces between the cement particles. These are electrostatic and steric push effects. The electrostatic impulse occurs due to the presence of a negative charge by the carboxyl groups and the steric repulsion effect is due to the long-edge polymers (Figure 1.a).

Dispersion Effect (Electrostatic effect)

The additive molecules are drawn by the soft cement granules and are wrapped around the cement during mixing. This formation increases the negative charges on the surface of the cement particles and causes the electrostatic impulse in Figure 2. This is the result of the large distribution of cement granules. This results in a significant increase in the workability of concrete, although the water content is low.

Steric Effect

Polycarboxylate-based additives have long-chain chains. They create a steric barrier. This increases the ability of cement particles to maintain their distance from each other and thus makes a perfect dispersion effect in (Figure 1.b).

A study investigated the effects of fly ash on the fluidity of the concretes produced by substituting fly ashes at 20%, 30%, and 40% ratios, and reported that fineness, density and zeta potential of fly ash affected the fluidity [6].

Another study investigated the rheological measurements of self-consolidating concretes (SCC) and found that the SCC obtained by adapting the Bingham model had very low shear stress values; in some cases, they were zero, and in some cases, they had negative values. The modified Bingham model was suggested instead of the Bingham model when the shear stress was negative [7].

In the study, the effect of high temperature on the physical and mechanical properties of mineral admixed self-consolidating concretes was found to be higher than the concrete with high strength and loss rate under high temperature and porosity increase rates due to the low structure of the self-compacting concrete with low admixture [8].

In the study, the effects of water/cement ratio, silica fume, limestone powder, additive type and amount on the compressive strength of high strength self-compacting concrete were investigated and it was observed that high strength self-compacting concrete with silica fume combined with chemical additive had a positive effect on compressive strength [9].
In this study, the effect of the addition of calcite and BFS on the performance of SCC was investigated. For this purpose, BFS has been replaced in place of cement in the production of SCC, while calcite is added in addition to cement.

**Materials and Method**

Materials and method used in the study are given in the below sections.

**Aggregate**

Limestone-based crushed stone with the largest grain size of 22 mm was used as aggregate in the study. The physical properties of the aggregates are given in Table 1 and aggregate sieve analysis are given Table 2.

**Table 1. Physical properties of aggregates**

| Physical properties | Fine aggregate | Coarse aggregate |
|---------------------|----------------|------------------|
| Sieve range         | 0-4 mm         | 4-12 mm          |
|                     | 12-22 mm       |                  |
| Density (g/cm³)     | 2.60           | 2.63             |
|                     | 2.65           |                  |
| Water absorption (%)| 1.50           | 0.75             |
|                     | 0.25           |                  |

**Chemical Additives**

For SCC study, polycarboxylate based super plasticizer chemical additive was used. The physical properties of the polycarboxylate based super plasticizing chemical additive used in the study are given in Table 3.

**Blast Furnace Slag and Calcite**

The blast furnace slag (BFS) used in the study was obtained from the 3rd furnace of Kardemir Karabük Iron and Steel Plant. The density of BFS was 3 g/cm³ and its specific surface area was 3785 cm²/g. The calcite obtained from Sivrihisar region and less than 75 μ sieve was used in the study. The density of the calcite was 2.72 g/cm³ and its specific surface area was 4160 cm²/g.

**Cement**

CEM I 42.5 R cement was used in the study. Chemical properties of cement are given in Table 4.

**Method**

In this study, the interaction between the chemical additive and the paste (cement, BFS, calcite, water, and chemical additive) was investigated in SCC in nano-micro, meso and macro levels. BFS at the ratios of 10%, 20%, 30%, and 40% was substituted with the arbitration specimens of cement, while 10%, 15%, 20% and 25% of calcite were added to the cement. Scanning electron microscopy (SEM) analysis of hardened pastes was performed at a micro level. The mini flow values of the meso-sized pastes were determined. The fresh state consistence, the specific bulk density, and the L-box test of the concretes prepared at a macro level were found, as well as the

**Table 2. Aggregate sieve analysis**

| Aggregate   | Last percentage (%) |
|-------------|---------------------|
|             | 0.125   | 0.25  | 0.5  | 1    | 2    | 4    | 8    | 16   | 22   | 32   |
| 0-4 mm      | 0.125   | 0.25  | 0.5  | 1    | 2    | 4    | 8    | 16   | 22   | 32   |
| 4-12 mm     | 0.00    | 0.15  | 3    | 60   | 90   | 100  | 100  | 100  | 100  | 100  |
| 12-22 mm    | 0.00    | 0.00  | 0    | 0    | 0    | 2    | 10   | 73   | 100  | 100  |

**Table 3. Physical properties of chemical additives**

| Properties     | Polycarboxylate |
|----------------|-----------------|
| Density (g/cm³)| 1.06            |
| pH             | 5.6             |
| Drymaterial content(%) | 25              |

**Table 4. Chemical properties of CEM I cement**

| Kimyasal özellikler | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | Na₂O | K₂O | SO₃ |
|---------------------|------|-------|-------|-----|-----|------|-----|-----|
| (%)                 | 21.16| 4.05  | 2.26  | 63.7| 1.30| 0.30 | 0.35| 3.30|
1, 3, 7, 28 and 90-day compressive strengths of hardened concrete samples. Table 4 shows the mixing ratios of the pastes. These pastes were placed into 5 mm diameter molds and SEM microphotograph images were obtained for hardened pastes for 28 days.

While preparing the pastes, the water/cement ratio was 0.33 and the additive ratio was kept constant at 1% of the cement weight. This ratio was taken for the full effect of calcite additions and BFS substitutions. When 0.50 water/cement ratio in the production of SCC was taken, the consistency was very fluid and the effect of calcite and BFS substitution was difficult to understand. For these reasons, it is necessary to fill the mini-slump funnel and the amounts given in Table 5 are taken to ensure a homogeneous mixture in the mixer. When these ratios were taken, the effect of calcite and BFS was clearly determined.

**Findings and Discussions**

The interactions at the nano-micro and meso levels of the prepared paste phase were evaluated, and its interaction at the macroscopic level was assessed based on the results of concrete experiments.

**Nano-micro Experiments**

When the pastes were prepared, mixing was carried out according to TS EN 197-1. Figure 2 shows that in SEM microphotograph analysis of BFS, large particles had smooth edge lines and smooth surface texture, while smaller particles had irregular and rough surface textures. Figure 3 shows that in the SEM microphotograph analysis, the calcite sample had more irregular geometric shapes, its surface texture was rougher, and its grain size distribution contained finer grains than BFS sample.

| Materials | Material Amount (g) |
|-----------|---------------------|
| Cement (g) | 1000 1000 1000 1000 |
| Water (g)  | 330 330 330 330 |
| Calcite (%) | 10 20 30 40 |
| BFS (%) | 10 20 30 40 |
| Additive (g) | 10 10 10 10 |

Table 5. Paste mixture amounts

Figure 4 shows the SEM microphotographs of hardened arbitration paste samples obtained on the days 1, 7 and 28. This analysis revealed that C-S-H gels were more intensively determined at the end of 28 days than other test aging days. In addition to the C-S-H gels, calcium hydroxide crystals were found in the medium. Also, in the analysis results of the aging day 1, highly unhydrated cement particles were observed in the medium.

Figure 2. SEM microphotograph of BFS, Figure 3. SEM microphotograph of calcite

Figure 4. SEM microphotographs of the arbitration specimen at different aging days
Figure 5 shows the SEM microphotographs obtained on the days 1, 7 and 28 for the pastes substituted with 60% Cement + 40% BFS and 25% calcite added. This analysis indicated that C-S-H gels were detected more intensively at the end of 28 days than other test aging days. Calcium hydroxide crystals were found to decrease. Calcite additions and microvoids were also observed to reduce. In the analysis results of the aging day 1, highly unhydrated cement and BFS were observed.

**Meso Level Tests**

Mini flow experiments were carried out using the pastes prepared for the experiments performed at meso level. Mini flow experiment was applied to prepared pastes. Flow measurement was performed by taking the mean distances measured from the farthest point of the paste and from the other endpoint perpendicular to this line (Figure 6).

Increased calcite addition amounts added to all paste mixtures resulted in reductions in mini flow values. The increase of the substitution amount of BFS led to higher mini flow values than arbitration samples. While the substitution ratios of BFS increased mini flow diameters, the calcite additions decreased them. The highest mini flow diameter was determined to be 246 mm in the paste mixture with 60% cement + 40% BFS + 10% calcite. The lowest mini flow diameter was measured to be 165 mm in the arbitration paste with 25% calcite (Figure 7).

**Macro-level Findings**

Macro-level concrete experiments were conducted. The cement dosage was taken as 350 kg/m3 for all prepared concretes. BFS was substituted with cement at the ratios of 10%, 20%, 30%, and 40%. Calcite was added to cement at the ratios of 10%, 15%, 20%, and 25%. The water/binder ratio was kept constant at 0.50 in all concrete mixtures. The effects of the BFS substitution and calcite additions on the consistency and process ability of the concrete were investigated. The consistency specific bulk density, and L-box test (the ability of the concrete to pass through reinforcements) were determined for the concrete in the fresh state (flow); and compressive strengths on days 1, 3, 7, 28 and 90 were determined for the hardened concrete samples.
Table 6 shows, the amount of mixture of SCC in 1 cubic meter is given.

Table 6
| Materials                  | Material Amount (g) (kg/m³) |
|----------------------------|-----------------------------|
| Cement                     | 350 315 280 245 210         |
| 0-4 aggregate              | 1000 965 930 895 860        |
| 4-12 aggregate             | 290 290 290 290 290         |
| 12-22 aggregate            | 570 570 570 570 570         |
| Water                      | 175 175 175 175 175         |
| Calcite (%10,15,20,25)     | 0 3 5 52 7 87 3 5 52 7 87 3 5 52 7 87 3 5 52 7 87 3 5 52 7 87 |
| BFS (%10,20,30,40)         | 0 35 70 105 140            |
| Polycarbosylate-based additive | 3,5 3,5 3,5 3,5 3,5         |
| Theoretical unit weight    | 2388,5 2388,5 2388,5 2388,5 2388,5 |
| Water/cement ratio         | 0,50 0,50 0,50 0,50 0,50    |
Figure 8 shows the passing ratios of the SCC produced in the study. Increased calcite additions were found to increase the passing ratios of the concretes. Because the calcite addition increased the amount of powdered material in the concrete, it facilitated the transport of concrete, its passage through reinforcements and homogeneous placement into the mold without any segregation. The highest passing ratio was found to be 0.85 in the paste mixture with 60% cement + 40% BFS substitution + 25% calcite addition. The lowest passing ratio was found to be 0.70% for the concrete with 60% cement + 40% BFS substitution.

As it is seen in Figure 9, decreases in spreading diameters were determined by increasing calcite addition rates. BFS substitution rates increased the spreading diameters.

Table 7 shows the consistencies consistency conservation, specific bulk densities, L-box passing ratios in the fresh state, as well as the compressive strengths of hardened concrete samples on days 1, 3, 7, 28 and 90. Concrete consistencies (flow) varied between 58 and 62 cm. After 30 minutes, SCC was found to retain their consistencies. The specific bulk densities of SCC were approximately 2400-2460 kg/m3. Although the increase in the amounts of calcite addition caused a decrease by 1 to 3 cm in the flow consistencies of SCC, a more homogeneous appearance and flow consistency were obtained. It was observed that the powdered materials more heavily packed and carried coarse aggregates as the amount of powder material increased. The BFS substitution ratio was found to decrease in early age compressive strengths. On day 1, the compressive strengths of 40% BFS substituted concrete samples reduced by approximately 100% compared to the arbitration specimen. However, a high strength transition occurred in BFS-substituted concrete for the days 28 and 90. It was determined that BFS had a positive effect on the strength of 28- and 90-day concretes.

Conclusion and Suggestions

Mini flow diameters increased with the increase in the BFS substitution ratios and decreased with the increase in calcite addition ratios.

It was found that the ability to pass through reinforcements increased with the increase in calcite addition. The highest passing ratio was found to be 0.85 in the paste mixture with 60% cement + 40% BFS substitution + 25% calcite addition. The lowest passing ratio was found to be 0.70% for the 60% cement + 40% BFS substitution. Calcite addition increased the volume of the powdered material in SCC and enabled the packing and transportation of the aggregates with a denser paste.

The SEM microphotograph analyses showed that the amount of calcium hydroxide decreased and the amount of C-S-H gels increased in the medium at the end of 28 days with the increase in the amount of BFS. As a result of the reaction of BFSs with calcium hydroxides, new C-S-H gels formed and the amount of calcium hydroxide in the hardened paste reduced.

The SEM microphotograph analyses also indicated that the increase of the calcite addition ratio increased the volume of the powder material and decreased microvoids.

The first days-strength values of concretes reduced by 90 to 100% with the increase in the BFS substitution ratio.

It was determined in the compressive strength tests of SCC at a macro level that BFS had a positive contribution to the compressive strength after 28 and 90 days.

Investigation and elimination of the negative effect of BFS on early aging will increase the usage rate of BFS in the concrete.

Flow Test

This test involved the observation of the deformation rate in fresh SCC using a flow table and the measurement of the diameter of the sample by flowing with its own weight, based on TS EN 12350-8 [10] (Figure 10). As the substitution ratio of BFS increased, compressive strengths decreased on day 1. However, the increased level was high at 28- and 90-day strengths. The highest 1-day compressive strength was obtained to be 18.97 MPa in the arbitration specimen + 20% calcite added to concrete. The lowest 1-day compressive strength was determined to be 6.74 MPa in 60% cement + 40% BFS + 5% calcite added concrete sample. While the compressive strength was low on day 1, the highest 90-day compressive strength was found to be 65.85 MPa in 60% cement + 40% BFS + 20% calcite added concrete sample. Figure 11 shows the L-box test setup. The experiment has been carried out according to Turkish Standard of TS EN 12350-10[8].
| Concrete Designs | W/C | Flow (t0) | Flow (t30) | Weight | L. Box passing ratio | 1 day | 3 day | 7 day | 28 day | 90 day |
|------------------|-----|-----------|------------|--------|----------------------|-------|-------|-------|--------|--------|
| Ref. Concrete    | 0.5 | 61        | 60         | 2435   | 0.76                 | 16.93 | 39.92 | 44.61 | 50.06  | 60.08  |
| REF + %10 C      | 0.5 | 62        | 60         | 2431   | 0.79                 | 18.41 | 43.45 | 52.88 | 58.81  | 61.28  |
| REF + %15 C      | 0.5 | 59        | 59         | 2422   | 0.79                 | 18.19 | 38.86 | 49.19 | 51.39  | 53.9   |
| REF + %20 C      | 0.5 | 58        | 57         | 2443   | 0.81                 | 19.87 | 45.12 | 50.45 | 55.65  | 57.26  |
| REF + %25 C      | 0.5 | 58        | 57         | 2465   | 0.81                 | 17.29 | 39.67 | 45.62 | 52.72  | 58.89  |
| %90 C + %10 BFS  | 0.5 | 60        | 61         | 2438   | 0.74                 | 16.33 | 34.67 | 41.85 | 49.88  | 57.15  |
| %80 C + %20 BFS  | 0.5 | 61        | 60         | 2418   | 0.72                 | 14.55 | 31.95 | 39.75 | 49.4   | 60.16  |
| %70 C + %30 BFS  | 0.5 | 62        | 62         | 2426   | 0.72                 | 12.19 | 28.97 | 38.27 | 49.88  | 61.33  |
| %60 C + %40 BFS  | 0.5 | 62        | 63         | 2433   | 0.70                 | 7.72  | 22.64 | 30.64 | 55.26  | 61.54  |
| %90 C + %10 BFS + %10 C | 0.5 | 59        | 59         | 2446   | 0.79                 | 17.32 | 38.16 | 44.56 | 55.41  | 61.59  |
| %90 C + %10 BFS + %15 C | 0.5 | 60        | 60         | 2421   | 0.80                 | 16.92 | 38.04 | 43.03 | 49.22  | 60.61  |
| %90 C + %10 BFS + %20 C | 0.5 | 58        | 58         | 2439   | 0.82                 | 17.18 | 37.18 | 42.95 | 53.47  | 61.1   |
| %90 C + %10 BFS + %25 C | 0.5 | 58        | 58         | 2451   | 0.84                 | 16.71 | 36.98 | 42.43 | 50.06  | 59.78  |
| %80 C + %20 BFS + %10 C | 0.5 | 61        | 62         | 2425   | 0.79                 | 11.11 | 29.65 | 39.78 | 49.16  | 61.93  |
| %80 C + %20 BFS + %15 C | 0.5 | 60        | 60         | 2442   | 0.80                 | 11.85 | 29.25 | 37.57 | 50.53  | 59.91  |
| %80 C + %20 BFS + %20 C | 0.5 | 59        | 60         | 2435   | 0.81                 | 13.2  | 28.69 | 40.4  | 47.46  | 62.48  |
| %80 C + %20 BFS + %25 C | 0.5 | 59        | 59         | 2419   | 0.83                 | 12.53 | 30.26 | 38.91 | 51.15  | 58.6   |
| %70 C + %30 BFS + %10 C | 0.5 | 62        | 62         | 2426   | 0.80                 | 11.59 | 26.46 | 35.62 | 50.51  | 61.72  |
| %70 C + %30 BFS + %15 C | 0.5 | 60        | 59         | 2424   | 0.80                 | 12.56 | 26.08 | 36.72 | 53.4   | 61.53  |
| %70 C + %30 BFS + %20 C | 0.5 | 60        | 61         | 2417   | 0.81                 | 10.32 | 25.86 | 32.59 | 43.73  | 60.61  |
| %70 C + %30 BFS + %25 C | 0.5 | 59        | 59         | 2431   | 0.83                 | 10.82 | 25.25 | 33.6  | 46.89  | 59.68  |
| %60 C + %40 BFS + %10 C | 0.5 | 61        | 60         | 2418   | 0.80                 | 7.95  | 20.64 | 32.69 | 55.39  | 63.03  |
| %60 C + %40 BFS + %15 C | 0.5 | 61        | 62         | 2433   | 0.82                 | 8.25  | 23.51 | 32.21 | 54.27  | 58.31  |
| %60 C + %40 BFS + %20 C | 0.5 | 59        | 60         | 2426   | 0.84                 | 9.32  | 26.1  | 34.96 | 53.61  | 65.85  |
| %60 C + %40 BFS + %25 C | 0.5 | 58        | 59         | 2425   | 0.85                 | 9.94  | 24.56 | 33.67 | 53.1   | 60.53  |

Table7. Compressive strength results of hardened concrete samples.
Figure 9. Flow diameters of fresh SCC

Figure 10. Flow diameter measurement of SCC

Figure 11. L – box test apparatus
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