



# Steel furnace slag aggregate expansion and hardened concrete properties



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## ABSTRACT

Steel furnace slag (SFS) is an industrial by-product that is not commonly utilized in bound applications because of its potential to contain high contents of free calcium and magnesium oxides, which expand when hydrated. In this study, a process was developed to quickly screen SFS aggregates for free oxide contents and expansion potential using complexometric titration, thermogravimetric analysis, and an autoclave expansion test. Two of the three SFS aggregate sources (high and low expansion) were selected for testing as a coarse aggregate in concrete. It was confirmed that SFS aggregates in concrete can produce acceptable strength properties, suitable freeze/thaw durability, and exceptional fracture properties. However, these SFS aggregates produced greater free drying shrinkage than concrete with dolomite aggregates. For SFS aggregates having low expansion potential, the hardened property tests indicate that SFS may be a suitable aggregate for concrete.

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## 1. Introduction

Steel furnace slag (SFS) is a waste by-product of steel production, with a worldwide SFS production of 150–230 million tons in 2012 [1]. Modern SFS is produced in either a basic oxygen furnace (BOF), where iron is converted to steel, or an electric arc furnace (EAF), where steel is produced by melting scrap steel [2]. BOF slag is also known as Linz-Donawitz (LD) or LD-converter slag. In these processes, calcite or dolomite fluxes are added to the molten steel in order to remove impurities, the result of which is called slag, consisting of a solid solution of various silicate and oxide mineral phases [2,3]. The impurity removal process that generates SFS introduces free calcium oxide (CaO) and/or free magnesium oxide (MgO) into the slag, both of which expand when hydrated.

While the composition of SFS depends on a number of factors, SFS typically has iron oxide contents greater than 20% by weight, has a lime-to-silica ratio greater than 7:3, and has low (<0.2%) sulfur contents [4]. The main mineralogical phases for both BOF and EAF slags are dicalcium silicate ( $2\text{CaO-SiO}_2$ ), dicalcium ferrite ( $2\text{CaO-Fe}_2\text{O}_3$ ), and wüstite (FeO) [5]. The free CaO contents typically range from 1% to 10% for BOF and 0% to 4% for EAF [6].

The increase in solid volume as free CaO and MgO hydrate to the hydroxide form are 91.7% for calcium hydroxide [ $\text{Ca}(\text{OH})_2$ ]

and 119.6% for magnesium hydroxide [ $\text{Mg}(\text{OH})_2$ ] [7]. These reactions are both topochemical, meaning that the hydroxide compounds evolve outward, causing stress concentrations that can lead to microcracking in confined or bound applications [7]. Since the expansion of SFS aggregates can approach 10% [8], SFS has not generally been used in confined or bound applications. Nevertheless, the hard and durable nature of SFS has made it a good candidate for use in asphalt pavements, railroad ballast, and fill material and for snow and ice control [9].

In North America, several forensic investigations of distressed SFS aggregate projects have been reported when used in concrete [10] and backfills [11,12]. In Europe, recent case studies have reported successful usage of SFS aggregates in concrete, such as for the Labein-Tecalia Kubik Building in Spain [13] and in concrete pavements and self-consolidating concrete in Greece [14,15].

Despite the potential for deleterious expansion, a number of laboratory studies have indicated that concrete with SFS can improve the concrete strength and modulus. Meanwhile, few studies have comprehensively characterized the SFS and examined the expansion potential along with the hardened concrete properties including durability. A number of concrete studies with SFS have shown acceptable strength performance and freeze/thaw and/or wet/dry durability [16–22], despite rather aggressive durability test conditions such as accelerated aging.

The main objectives of this study were to examine the chemical and mineralogical composition, expansive potential, and free oxide content of three different SFS sources. Additionally, several concrete mixtures were batched using SFS aggregates to characterize the properties of concrete containing SFS as well as compare the

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effects on the strength, fracture, shrinkage, and freeze/thaw durability properties of concrete.

## 2. Materials and testing methods

### 2.1. Aggregate materials

The three SFS coarse aggregates evaluated were BOF, EAF, and EAF/LMF. In a ladle metallurgy furnace (LMF), supplemental alloying or fluxing agents can be added to the molten steel after the EAF processing to ensure proper chemistry of the final steel product. As a control mix, crushed dolomite coarse aggregate was also evaluated. Natural sand was used as the fine aggregate for all mixtures. The evaluated aggregate physical properties included gradation (ASTM C136 [23]), specific gravity (ASTM C127 and C128 [23]), and unit weight (rodding method) (ASTM C29 [23]).

### 2.2. Chemical and mineralogical characterization

Powder X-ray diffraction (XRD) was utilized to determine the mineralogical composition of the SFS. A Siemens–Bruker D5000 XRD was used, which utilizes copper (Cu)  $K\alpha$  radiation and has a graphite monochromator and a scintillation detector. The scan range was from  $10^\circ$  to  $80^\circ$  with an increment of  $0.02^\circ$  and a scan speed of 0.5 degrees per minute. The SFS samples were ground and particles passing the #100 sieve ( $\leq 150 \mu\text{m}$ ) and #325 sieve ( $\leq 44 \mu\text{m}$ ) were collected for analysis.

Inductively coupled plasma (ICP) spectroscopy has been a technique that has been routinely applied for quantitative elemental analysis of SFS [24–31]. In this study inductively coupled plasma optical emission spectroscopy (ICP-OES) was conducted, using a PerkinElmer Optima 2000DV ICP-OES. The tested sample had particles that were all passing the #100 sieve ( $\leq 150 \mu\text{m}$ ). The composition detection was limited to only the main elements in typical SFS samples, namely iron, calcium, silicon, magnesium, manganese, aluminum, titanium, sulfur, phosphorus, and chromium.

Ethylene glycol and complexometric titration has previously been used to determine the free CaO content of cement and clinker [32], but it is also been applied to SFS [5,27,33–39]. Ethylene glycol will form a complex with any free CaO present, but will also complex with some, but not necessarily all, of the  $\text{Ca}(\text{OH})_2$  present [40]. Adding phenolphthalein as an indicator, the solution can then be titrated with hydrochloric acid (HCl) to determine the calcium content. The ethylene glycol number (EGN), which represents the total CaO that is dissolved from the free CaO and free  $\text{Ca}(\text{OH})_2$  in the sample, is determined as a percentage, following Eq. (1), where  $m$  is the initial mass in grams of SFS sample,  $N_{\text{HCl}}$  is the normality of the HCl,  $V_{\text{HCl}}$  is volume in mL of HCl titrated,  $V_{\text{blank}}$  is a correction for the volume in mL of HCl titrated in a blank ethylene glycol sample (which was found to be zero, since ethylene glycol has a relatively neutral pH), and  $F$  is an equivalency factor. The equivalency factor  $F$  for this method and equation is 28 [41].

$$\text{EGN} = F \left[ \frac{N_{\text{HCl}}(V_{\text{HCl}} - V_{\text{blank}})}{10m} \right] \quad (1)$$

Since the ethylene glycol also dissolves  $\text{Ca}(\text{OH})_2$ , thermal analysis can be used in conjunction with complexometric titration to better refine the free CaO content determination of SFS [31,33,36,38,39,42,43]. For this study, thermogravimetric analysis (TGA) was conducted to determine the hydroxide and carbonate contents in the SFS. A TA Instruments Q50 TGA was utilized in this analysis, which heated a 20 mg powdered SFS sample to  $1000^\circ\text{C}$  ( $1830^\circ\text{F}$ ) at a heating rate of  $10^\circ\text{C}$  ( $50^\circ\text{F}$ ) per minute. Nitrogen was used as the purge gas at flow rates of 60 mL/min for the

sample purge and 40 mL/min for the balance purge. The nitrogen environment was selected to prevent carbonation of the free oxide and hydroxide phases in the sample.

### 2.3. Expansion testing

Conventional expansion testing for aggregates is specified in ASTM D4792. Previous testing has shown that SFS aggregates can undergo greater expansion than natural aggregates [44]. However, since MgO hydration may take longer than the 7-day minimum expansion recommended in ASTM D4792, the testing protocol in this study instead utilized an autoclave expansion test in which a sample was subjected to steam at 300 psi (2.1 MPa) and  $420^\circ\text{F}$  ( $216^\circ\text{C}$ ) for three hours, similar to the ASTM C151 autoclave test for cement. This test method was adapted from the procedure developed by Edw. C. Levy Co. (J. Yzenas, personal communication, 2014). Since previous studies have shown that the aggregate gradation can affect the expansion of SFS [45,46], the material was sieved and re-graded (Table 1) in order to directly compare the expansion of the different SFS samples with similar gradations. Additionally, a “monoparticle” size gradation was tested, where only particles passing the 1/4 in. (6.35 mm) sieve and retained on the #4 (4.75 mm) sieve were used.

The mold assembly into which the aggregate sample was compacted, seen in Fig. 1, consisted of a specimen mold affixed to a base plate. The specimen mold was 3.1 in. (7.9 cm) in diameter and 2.3 in. (5.8 cm) in height. An extension collar was connected to the specimen mold, where a stem and surcharge are placed on top of a compacted sample. The weight of the stem plus the surcharge was 3.22 lb (1460 g). The stem was perforated to allow steam to enter the sample. The compaction of the specimen was performed using a 5.50-lb (2.5 kg) hammer dropped from a height of 12 in. (30 cm). The aggregate was compacted in three lifts with 25 blows per lift. A straightedge was used to trim the excess material from the mold in order to ensure a constant initial specimen height. The aggregates were oven-dried prior to compaction.

The height of the stem on top of the sample was measured in inches before ( $h_i$ ) and after ( $h_f$ ) autoclaving using a dial gauge fixed to a stationary stand. This height was also measured relative to a reference specimen of constant length before ( $\text{ref}_i$ ) and after ( $\text{ref}_f$ ) the sample the autoclaved. As shown in Eq. (2), the percent expansion ( $e$ ) was determined relative to the gauge length ( $G$ ) of the sample, which was 2.3 in. (5.8 cm).

$$e = \frac{(h_f - \text{ref}_f) - (h_i - \text{ref}_i)}{G} 100\% \quad (2)$$

### 2.4. Concrete properties

An average of three specimens was tested for the compressive and split tensile strengths (AASHTO T22 and T198 [47]), free shrinkage (AASHTO T160 [47]), and freeze/thaw durability (AASHTO T161 [47]). The strengths were evaluated using 4 by

**Table 1**  
Target dense gradation for autoclave expansion test.

Passing	Retained on	Mass percent (%)
1/2 in. (12.5 mm)	3/8 in. (9.5 mm)	14
3/8 in. (9.5 mm)	1/4 in. (6.35 mm)	17
1/4 in. (6.35 mm)	#4 (4.75 mm)	10
#4 (4.75 mm)	#8 (2.36 mm)	21
#8 (2.36 mm)	#16 (1.18 mm)	14
#16 (1.18 mm)	#30 (0.6 mm)	9
#30 (0.6 mm)	#50 (0.3 mm)	8
#50 (0.3 mm)	#100 (0.15 mm)	4
#100 (0.15 mm)	#200 (0.075 mm)	3

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