The effects on the properties of self-compacting mortar of using stainless steel blast furnace slag as a sustainable nano material addition

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Abstract. Voids in cementitious mortar materials cause a drastic reduction in the load carrying capacity of the element. Furthermore, weak interfaces between cement paste and the aggregate creates micro crack initiation and crack propagation. It is therefore very important to decrease these pores and enhance the microstructure homogeneity of cement mortar by adding nano particles. In this article, the properties of self-compacting mortar containing nano blast furnace slag were experimentally assessed. Nano blast furnace slag was prepared and used as an additive for self-compacting mortar in 1, 2, 3, 4, and 5% by weight of cement proportions to generate cementitious sustainable materials. Blast furnace slag was crushed and milled to nano particle size, as checked by Laser Particle Size Analysis, AFM, and FESEM. The self-compacting mortar properties were then tested at 7, 28, and 90 days age. The flowability of self-compacting mortar was determined using the mini flow table and mini v-funnel tests. The experimental results for the hardened mortar showed that the modification and enhancement of the mechanical properties, including compressive strength, flexural strength, and direct tensile strength, increased as the nano powder percentage increased. The total water absorption and porosity were also decreased as the nano powder percentage increased, while the density of self-compacting mortar was decreased. The microstructure homogeneity and nanoparticle distributions of self-compacting mortar were also examined using FESEM.

1. Introduction
Self-compacting concrete (SCC) is considered a promising concrete technology that is advancing rapidly. The self-compatibility of SCC is obtainable by limiting the coarse aggregate and maximum size and using lower water/powder ratios simultaneously with the addition of superplasticizers [1]. Thus, the workability properties mainly depend on the material’s components, and the segregation and bleeding of self-compacting concrete can be prevented by increasing the fine aggregate powder content and the use of viscous modified admixtures [1]. Mortar acts as the essential part responsible for the workability properties of self-compacting concrete, and these properties can thus be assessed in self-compacting mortar [2]. Self-compacting mortars, as a part of multiple concrete technology products, are preferred for the rehabilitation and repairing of reinforced concrete structures [3]. Numerous materials with cementitious and pozzolanic characteristics are thus used as replacement materials in cement for concrete productions. Some of these materials are industrial wastes or by-products, such as fly ash, slag, and silica fume [4,5].
Blast Furnace Slag (BFS) is defined as “non-metallic products, which includes basic oxides such as silicate and alumina silicate of calcium and others... generated in a molten condition concurrently with iron in a blast furnace” by ASTM C 125-16 [6], and it is widely used in the construction industry. Slag includes multiple steelmaking by-products formed during the processing of pig iron by basic oxygen or electric arc furnace steelmaking. There have already been a significant number of studies on reducing the environmental effects of the steel industry and finding different ways to recycle materials and thus ensure its economic sustainability [7]. A summation of the most research results on the use of nanoparticles for cementitious materials indicates that the compressive and flexural strength of cement mortar in the presence of nano- SiO$_2$ and nano-Fe$_2$O$_3$ sees a significant increase as compared to conventional mortar mixed with the same w/c ratio, as the nanoparticles enhance the hydration process. These mixes thus exhibit the best cement-paste microstructure quality [8-10]. On the other hand, the use of nano-SiO$_2$ results in a more effective pozzolanic reactions when compared to the use of silica fume, as the nano-SiO$_2$ not only functions as a development agent for the microstructure of the mortar but also as an activator for the pozzolanic reaction [11,12].

The main objective of the current paper is thus to investigate the influence of nano blast furnace slag when used as an additive by weight of cement in the fresh state and the resultant hard state properties of the self-compacting mortar produced.

2. Experimental programs
Three stages were undertaken achieve the aims of this study: the preparation of nano blast furnace slag, the assessment of self-compacting properties using flow diameter and flow time tests, and the assessment of the mechanical and physical properties of the hardened mortar at 7, 28 and 90 days of curing. The microstructure homogeneity was also examined and explained.

2.1. Materials used
Ordinary Portland cement (OPC) Type I conforming to ASTM C150[13] was used; the chemical components and physical properties of Portland cement and nano blast furnace slag are given in tables 1 and 2 respectively. Micro silica powders were used as a Pozzolanic admixture, conforming to the requirement of ASTM C-1240[14], with a specific gravity equal to 2300kg/m$^3$, SiO$_2$ greater than 85%, SO$_3$ less than 2%, fineness larger than 15000 m$^2$/kg, and an activity index of 147%. Natural river sand was used as a fine aggregate with a maximum size of 4.75mm; its specific gravity, fineness modulus, sulphate content, clays and fine material, and absorption capacity were 2.65, 2.61, 0.086 %, 2.9%, and 1.75%, respectively. The grading and the characteristics of the sand conformed to ASTM C33[15]. A polycarboxylic ether, a modified high range-water reducer admixture conforming to ASTM C-494 type F&G [16] was also used with a relative density of 1.07.

| Table 1. Chemical components of Portland cement and Nano blast furnace slag. |
|---------------------------|--------------------------|------------------------|
| Components | Cement Type I [Wt.%] | Nano blast furnace slag [wt. %] |
| CaO | 61.11 | 0.7 |
| SiO$_2$ | 20.38 | 33.59 |
| Al$_2$O$_3$ | 5.82 | 32.06 |
| Fe$_2$O$_3$ | 3.28 | 21.62 |
| MgO | 4.27 | / |
| MnO | / | 6.9 |
| SO$_3$ | 2.12 | 0.03 |
| Other oxides | / | 5.03 |
Table 2. Properties of Portland cement used.

| Physical properties                          | Results | Limits of ASTM |
|---------------------------------------------|---------|----------------|
| Specific surface (m²/kg).                   | 395     |                |
| Setting time (Vicat’s method)               |         |                |
| -Initial setting (min)                      | 90      | ≥ 45           |
| -Final setting (min)                        | 210     | ≤ 375          |
| Compressive strength of Mortar (MPa)        |         |                |
| 3-days                                      | 20.3    | ≥ 12           |
| 7-days                                      | 30.1    | ≥ 19           |
| Expansion (Autoclave), Max,%                | 0.05    | ≤ 0.8          |
| L.O.I. Max,%                               | 2.16    | 3              |
| L.S.F. %                                    | 0.88    |                |
| I.R. Max, %                                 | 0.36    | 0.75           |

2.2. Production of nano blast furnace slag (BFS) powder

BFS is a by-product of the iron industry, in this case obtained from Al Nasr Facility, Bagdad, Iraq. This is an amorphous, coarse material that when finely chopped and incorporated with cement exhibits hydraulic cementitious features. Although the average granular size of blast furnace slag differs by source, and the chemical composition depends on the composition of raw materials, the main constituents of blast furnace slag are SiO₂, Al₂O₃, CaO, and MgO which cover 95% of the composition [18]. Nano blast furnace slag was prepared here by using a jaw crashing process to minimise the particle size of the slag, which was then sieved to pass from a 2.36 mm sieve size. A planetary stainless-steel ball mill instrument (PBM) was then used to produce powder. The stainless steel ball that undergoes rotational movement within the PBM container comes into impact with the inner wall of the milling bowl [17], and the duration of the milling process was about five hours for one kilogram of stainless steel blast furnace slag, as shown in Figure 1. Laser particle size analysis, atomic force microscopy (AFM), and field emission scanning electron microscope (FESEM) processes were used to assess the size of the nanoparticles of blast furnace slag powder achieved. The results as shown in Figure 1 indicated that nanoparticle scale was achieved. Laser particle size analysis and AFM tests also indicated that the average diameter of the nanoparticles of slag powder were 67.5nm and 64.46nm respectively, while the FESEM gave the nanoparticle size and the shape of nano blast furnace slag powder particles as being within the nanoscale. The activity index results for blast furnace slag were equal to 71.8 % at 7 days, and 93.2 % at 28 days, allowing it to be classified as Grade 100 according to ASTM C989 – 04 [27].
3. Mix proportion
Details of the mix proportions for self-compacting mortars containing nano blast furnace slag powder are given in table 3. The cementitious water ratio (W/CM) was 0.35, and five percent nano blast furnace slag powder was added by weight of cement and mixed with water via an electric mixer. In all mixtures, the amount of superplasticizer used was such that no segregation and no bleeding was reported.

Table 3. Mix proportions of self-compacting mortar with/without nano blast furnace slag.

| Mix ID  | Ref. mix | NBFS1 | NBFS2 | NBFS3 | NBFS4 | NBFS5 |
|---------|----------|-------|-------|-------|-------|-------|
| Nano slag % | 0 | 1 | 2 | 3 | 4 | 5 |

4. Tests methods

4.1. Fresh mortar tests

4.1.1. Mini Slump. Cone mould apparatus was used to determine the slump flow diameter of the self-compacting mortar. The cone mould dimensions were 100 mm at the base, 70 mm top diameter, and 60 mm high. The cone was placed at the centre of the steel base plate and filled with mortar before
being upwards to allow the mortar to spread without any segregation and bleeding over the table; the subsequent diameter of the mortar was measured in two perpendicular dimensions and the average was reported as final diameter (mm) of spread mortar. This test was completed according to EFNARC 2005 [19], ASTM C1437 – 03 [20], and ASTM C230M-03 [21].

4.1.2. Mini V-Funnel Flow. A suitable water/binder ratio of the mixture was selected via v- funnel testing: the apparatus was filled up with a mortar, and then the gate was unlocked and a stopwatch started. The watch was paused when light first appeared when looking down into the funnel from above; the flow time (in sec.) was then recorded, according to the procedure outlined in EFNARC 2005 [19].

4.2. Hardened mortar tests
Three average hardened mortar specimens tests were adopted to examine each property; the compressive strength was tested according to ASTM C109/C109M-12[22], flexural strength according to ASTM C348 [23], direct tensile strength according to ASTM C190 – 85 [24], and dry density, total absorption, and total porosity properties according to ASTM C642 – 13 [25].

5. Results and discussion

5.1. Fresh properties
The slump flow diameter and flow time of self-compacting mortars are presented in table 4. The results reflect the viscosity and segregation resistance of the mortar mixtures. The slump flow diameters of all mixtures were in the range of(260 to 283 mm, and flow times were in the range between 8 and 13 Sec. Thus, the fresh mixtures’ workability properties conformed with EFNARC [19] recommendations. Furthermore, the self-compacting mortar flowability estimated by using mini v-funnel, based on the time of the mortar mixture required to flow through the funnel, showed that the increase in nano blast furnace slag powder percentage led to increases in flow diameter and reductions in the flow time due to the larger specific surface area and small particle size of the nanoparticle powders, offering more filling ability and mortar flowability. Generally, mineral admixtures such as blast furnace slag improve the workability of fresh mortar, creating a smooth and dense surface that allows smooth slip [18].

Table 4. Mini flow table and Mini v-funnel tests results.

| Mix ID       | Ref.Mix | NBFS1 | NBFS2 | NBFS3 | NBFS4 | NBFS5 |
|--------------|---------|-------|-------|-------|-------|-------|
| Mini-Slump Test (mm) | 259.17  | 265.2 | 270.2 | 276.7 | 281.63| 283.2 |
| V-Funnel Test (sec.) | 13   | 12    | 10.5  | 9     | 8.5   | 8     |

5.2. Physical properties
Dry density, water absorption, and total porosity tests of self-compacting mortar were carried out at 7, 28, and 90 days of curing. The total porosity of the mortar mixes was obviously affected by the use of nano stainless-steel blast furnace slag, which could be effectively used to reduce the pore and accumulative pore volume, leading to more impermeable mortars. As shown in Figure 2, the dry density of hardened mortar increased with the addition of nanoparticle powder and densified with curing time as compared with the reference mix, while water absorption and total porosity results as shown in Figure 3 and Figure 4, respectively, indicated that the water absorption and total porosity decreased as nanoparticle powder additive percentage increased. Water absorption is generally related to the structure of the pores and porous paste’s aggregate interface zone [28].
5.3. Mechanical properties

The mechanical properties, including compressive strength, direct tensile strength, and flexural strength, of self-compacting mortar were developed by the addition of nanoparticle powders. The results in Figure 5 show that the compressive strength increased with the addition of nano stainless-steel blast furnace slag powder as compared with the reference mortar mix specimen. Compressive strength increases were noticed for all curing ages, and the direct tensile strength, as seen in Figure 6, and the flexural strength as seen in Figure 7, also improved with the addition of nanoparticle powders at 7, 28, and 90 days. Mechanical properties’ enhancement in cement mortar composites due to the use of nanoparticles has been observed by others [26,17]. The high chemical reactivity of nanoparticles is beneficial for creating additional nucleation places that permit formation of more C-S-H phases, which have an effect on concrete strength. In the case of nano blast furnace slag, which contains a mixture of SiO$_2$, Al$_2$O$_3$, Fe$_2$O$_3$, and other oxides, the smaller slag particles which have both cementitious and pozzolanic properties, offer better hydration processes and create smaller ettringites and calcium hydrates. Nano additives, due to their fine particle size and nature, can also increase the packing density of a matrix, while the chemical reactions between the siliceous nanoparticles and calcium hydroxide lead to the formation of more C-S-H in microstructures. The worst mechanical properties are related to the agglomeration of nanoparticles and formations in weaker areas within the hydrated binder matrix [29].
5.4. Microstructure

Self-compacting mortar containing nano stainless-steel blast furnace slag is less porous, with smaller pore size and densify microstructure, than the reference mortar. The homogeneity of the hardened mortar microstructure can be observed by field emission scanning electron microscope analysis. Nanoparticles powder acts as filler or binder that enhances the performance of interfacial transition zones between the aggregates and cementitious materials matrix as well as increasing the quality of the cement matrix itself [17]. The blast furnace slag and Portland cement work together with water to form calcium silicate hydrates. Furthermore, the nanoparticle powder reacts with excesses of calcium hydroxide to form a finely dispersed gel, which fills large pores, and, as a result, the hardened cement paste contains fewer calcium hydroxide crystals and therefore has fewer large capillary pores. Further, the distribution of nanoparticle powder allows the creation of identical products with a better microstructure of hardened mortar, as noted in Figure 8.
Figure 8. Particle distribution inside the microstructure of self-compacting mortar: (A) without nanoparticle slag powder, (B) self-compacting mortar with 3% additive nanoparticle slag, and (C) self-compacting mortar with 5% additive nanoparticle slag. L1, L2, and L3 clarify the nanoparticle size.

6. Conclusions
The test results from this study allow the following conclusions to be drawn:

a. Nanoparticles of stainless-steel blast furnace slag powder show cementitious and pozzolanic activity when used as a powder addition to conventional cement mortar, with fineness affecting the reactivity properties of the slag in the cement mortar. The related increase in the specific surface area leads to better strength development.

b. The addition of stainless-steel blast furnace slag powder to mortar mixes leads to
   1. Increases in the dry density by about 0.4% to 1.2% and 0.14% to 1.4% for mortar at 28- and 90-days age, respectively.
   2. Reductions in the percentage of water absorption and total porosity of about 7.33% to 36.36% and 38.3% to 45 %, and 0.35% to 25.3% and 33.33% to 41.17% at 28- and 90-days age, respectively.
3. Increases in the compressive strength, direct tensile strength, and flexural strength by about 2.8% to 28.5% and 6.4% to 35%; 1.8% to 34.6% and 0.5% to 30.8%; and 2.5% to 9.6% and 4.55% to 13.5%, respectively, compared with conventional reference mortar at 28- and 90-days age respectively.

4. Densification of the microstructure of the matrix.

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