Effect of Microsilica on Strength and Microstructure of the GGBS-based Cement composites

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Abstract. Cement production requires substantial energy and significantly accounts for global carbon dioxide emissions. Thus, the consumption of ordinary portland cement (OPC) must be reduced through incorporation of auxiliary materials. Also, the mechanical strength and durability of structures, in the construction industry, need to be improved for economizing the maintenance cost and increasing the service life. This study explores the effect of partial substitution of cement by granulated blast furnace slag (GGBS), and microsilica (MS), the industrial by-products. This study focuses on the use of 10% GGBS and 0-16% MS as a substituent of cement at a water binder ratio of 0.42. The fresh properties were determined to study the effect of these substituents. The compressive strength of all the mixes was determined after 3, 7, 28, and 56 days of treatment. The results were correlated through microstructural analysis. The study revealed that the cement composites with an optimal substituent dosage of 10% GGBS and 12% MS can attain adequate compressive strength and can be used for practical applications.

1. Introduction

Construction technology can aid in sustainable development by using less energy and natural resources along with the production of lesser waste material. The concern also focuses on the improvement of fresh as well as hardened properties of the mortar. The quality of cement mortar is characterized by its mobility, retention of water, strength, and durability and depends on its ingredients [1]. A good mortar should be economical, easy to work, well-adherent, durable, and chemical attack resistant. As per ideal requirements, the paste should not only be workable, but the constructed material should be durable with high mechanical strength. Particle size distribution (PSD) controls the flow of mortar, while the mechanical properties and durability are controlled by the mix grading and the resultant packing in the matrix. Further, the hydration behavior and the requirement for flow is dependent upon the extent of cement dispersion in water[2]. The flocculation level of a cement paste is often related to water content and early hydration. PSD, matrix packing, and water demand in cement mortar can be controlled by using admixtures such as water-reducing compounds and mineral admixtures [3].

Sustainable development is described as the development that meets the needs of the present generation without sacrificing next-generation needs. The production of the prime constituents of cement mortar i.e. cement and fine aggregates pose an environmental threat. Likewise, the use of an enormous amount of drinking water leads to scarcity of these resources in the long run [4]. As per
estimation, one ton of cement clinker production yields one ton of greenhouse gases (GHGs) such as carbon dioxide responsible for global warming. The production of fine aggregates requires extraction from quarries at the expenditure of energy with the emission of waste products leading to environmental pollution. Thus, the construction industry is not only one of the largest consumers of energy and natural resources but also an immediate concern for environmental issues [5]. By incorporating different mineral admixtures such as metakaolin, GGBS, fly ash, rice husk powder, palms oils, and silica fumes, concrete consistency can be improved. The hardness of concrete is also influenced by mineral admixtures. The integration of these admixtures reduces cement content which reduces the environmental impact and also improves the properties of concrete [6]. Waste disposal problems can also be reduced as they are industrial by-products. An improvement in the mechanical properties and toughness of cement indicates the positive influence of mineral mixtures on concrete [7]. The use of these additional cementing materials in concrete preparation will lead to substantial improvements in resources, cost efficiency, and a decrease in environmental emissions [8].

Microsilica (MS) has been found to enhance the physical and mechanical aspects of cement mortar to a great extent [9]. MS is an important pozzolanic and cementitious material consisting of very fine and non-crystalline silica obtained by the processing of silicon-containing alloys or elemental silicone in electric arc furnaces [10]. MS particles have an average microsphere diameter, of roughly 0.15 microns, and primarily belongs to the cristobalite, vitreous form of silica. On average, one microsphere weighs 100 times lesser than a typical grain of cement. Owing to its fine quality and rich content of silicon dioxide, it plays an important role in pozzolanic action in the cement matrix (Equation 1 and 2).

$$\text{2SiO}_2 + 3\text{Ca(OH)}_2 \rightarrow 3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O} \quad \text{(Equation 1)}$$

$$\text{2SiO}_2 + 3\text{CaO} + 3\text{H}_2\text{O} \rightarrow 3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O} \quad \text{(Equation 2)}$$

Microsilica results in enhancement of CSH gel production and refined pore structure, thereby reducing permeability and improving the durability of cement mortar [11]. It also results in a hindrance to the diffusion of detrimental ions and increases the resistance towards an aggressive environment [12].

Ground granular blast furnace slag (GGBS) is a mineral admixture, generally extracted from molten slag as a granular substance that is grounded after trying to get a powdery form. GGBS has pozzolanic and cementitious properties [13]. GGBS has unique characteristics in terms of resistivity to corrosion, attack of sulfate, and permeability of water. GGBS reduces creep and bleeding but increases the heat of hydration, resulting in improved compressive strength of the mortar. GGBS is an aluminosilicate with main constituents as SiO$_2$, Al$_2$O$_3$, CaO, and MgO [14]. The addition of GGBS was found to improve the workability and the long-term compressive strength [15]. The incorporation of SF and granulated blast furnace slag was found to provide lower early age strength to coral aggregate-concrete but higher late age strength due to late initiation of the pozzolanic reaction [16]. Auxiliary addition of GGBS and SF was found to boost the mechanical characteristics of fly ash-based concrete [17]. The combination (MS 10%+ GGBS 30%) was found to provide a more workable and higher strength relative to standard concrete for all curing days [18]. The replacement of cement by SF and GGBS in presence of fly ash and metakaolin was found to increase the consistency and retard the setting time [19]. The optimal dosage of SF and GGBS has been obtained as 10% for the best workability and strength of concrete [20]. The incorporation of 10% SF and 10% GGBS was found to provide maximum compressive strength and abrasion resistance of concrete [21]. Self-compacting concrete was found to be more cost-effective and with better strength with the incorporation of GGBS, SF, and metakaolin [22]. The presence of auxiliary GGBS and SF was also found to improve the compressive strength of otherwise weak recycled aggregate-based concrete [4].
In this study, the effect of partial substitution of cement with MS in presence of GGBS was analyzed on the fresh properties, strength, and microstructure of cement mortars at primitive and advanced ages. GGBS was incorporated at 10% by weight of cement as a partial substituent of cement. MS content was varied as 4%, 8%, 12%, and 16% by weight for partial substitution of cement. The microstructural analysis of cement mortars with and without MS was carried out using Scanning Electron Microscope (SEM) studies.

2. Materials and Methods
The cement used for the experimental studies was ordinary Portland cement (43 Grade). GGBS and Micro-silica were obtained as a by-product of the local industry and was used as such without any modification. The Chemical composition of the binder materials have been given in Table 1. Fine aggregates were procured from the locally available source and conformed to IS: 383-1970 [18]. The physical characteristics of components of mortar have been given in Table 2. 10% GGBS was used as a fixed substituent by weight of cement for all the mixes, except the control mix (CM). Besides, cement was substituted partially by 4%, 8%, 12% and 16%MS by weight of cement as per mix composition given in Table 3. The consistency and setting time of the cement was determined through Vicat's Apparatus and flow table was used for flow determination of mortars [19]. The mortar was prepared with a binder: sand ratio of 1:3 with a water-binder ratio at 0.42. The components were mechanically mixed and moulded into cubes with proper compaction to eliminate furthermore air voids. The mortar cubes were allowed to set for 24 hours and then placed in a water tank for curing at standard temperature. Compressive strength is a key value for construction practices. The compressive strength of the fully cured, surface dried cement mortar specimens was measured with a compression testing machine at 3 days, 7days, 28 days, and 56 days. Testing was carried out with three cubes and the average value was considered [18]. Microstructural analysis was carried out through Philips XL20 Scanning Electron Microscope.

| Table 1. Chemical Composition of Binder Materials |
|-----------------------------------------------|
| Oxides | OPC | MS | GGBS |
| % by mass | % by mass | % by mass | % by mass |
| SiO₂ | 19.46 | 98 | 35.6 |
| Al₂O₃ | 4.22 | 0.03 | 13.19 |
| Fe₂O₃ | 3.56 | 0.02 | 1.13 |
| CaO | 65.92 | 0.3 | 41.94 |
| MgO | 1.08 | 0.2 | 3.76 |
| K₂O | 0.67 | 0.3 | 0.41 |
| Na₂O | 0.21 | 0.3 | 0.32 |
| TiO₂ | 0.24 | - | 0.52 |
| SO₃ | 2.6 | - | 1.5 |
| Cl | 0.01 | - | 0.01 |
| LOI | 0.96 | 0.8 | 1.41 |

| Table 2. Physical Properties of Components |
|-------------------------------------------|
| Aggregate Type | Bulk Density (kg/m³) | Fineness (m²/kg) | Specific gravity |
|----------------|-----------------------|------------------|-----------------|
| GGBS          | 1000-1100             | 350-550          | 2.90            |
| MS            | 1420-1540             | 15000-35000      | 2.22            |
| Fine aggregate| 1615                  | 1.15             | 2.55            |
Table 3. Mix proportion of Specimens per m³

| Designation | Cement (g) | Sand (g) | GGBS (g) | MS (g) | Water (g) |
|-------------|------------|----------|----------|--------|-----------|
| CM          | 200        | 600      | -        | -      | 84        |
| MS1         | 172        | 600      | 20       | 8      | 84        |
| MS2         | 164        | 600      | 20       | 16     | 84        |
| MS3         | 156        | 600      | 20       | 24     | 84        |
| MS4         | 148        | 600      | 20       | 32     | 84        |

3. Results and Discussions

3.1. Fresh Properties

The observed values of consistency of mortars, without and with the replacement of cement with GGBS and MS have been plotted in figure 1. The consistency of mortars increased with the incorporation of GGBS and with an increasing percentage of MS from 4% to 16%. The consistency of the mortar is related to the size and specific surface of the particles present in the mortar [8]. The microparticles of GGBS and MS have a greater specific surface of the particles. As the percentage of MS increases, the hydration process is faster and the water required for the hydration increases, increasing the consistency of mortars [19].

Figure 2 illustrates the effect of GGBS and MS on the setting time of mortars. Initial setting time (IST) as well as final setting time (FST) of mortars, increased with substitution of cement with GGBS, and with an increasing percentage of MS from 4% to 16%. The retardation effect has been attributed to the decreasing content of cement with the auxiliary GGBS and MS. The setting time is related to the extent of hydration reaction in the mortar. The literature reports the delay of hydration reaction by GGBS [19]. The fine particles of MS fill the voids in the matrix, hence, the porosity of the mortars decreased and the setting process gets decelerated, resulting in an increase in setting time of mortars [23]. The observed values of the flow of mortars, without and with the replacement of cement with GGBS and MS have been plotted in figure 3. The flow of mortars was found to decrease with auxiliary GGBS and with an increasing percentage of MS from 4% to 16%. The flow of the mortar is also related to the size and specific surface of the binder particles [24]. The fine particles of GGBS and
MS have a greater specific surface of the particles and result in better particle size distribution in the mixture. The filler effect of these particles reduces the pores and improve the packing of constituents in the matrix. As the percentage of MS increases, cohesive binding results in enhanced packing and the empty spaces in the matrix are better filled. As a result, water cannot bleed over the surface of the matrix. This increases the stiffness of the mortars resulting in a decrease in the flow of mortars. Similar effects have been reported in the literature [20].

3.2. Compression Strength
The effect of partial substitution of cement by GGBS and MS on the compressive strength of mortar at the age of 3, 7, 28, and 56 days has been analyzed in comparison to CM and represented in figure 4. The compressive power of all mortar mixtures, MS1, MS2, MS3, and MS4 at 3 days were higher
compared to CM. Nevertheless, it has been observed that the compressive power of mortars has improved with substitution by GGBS as well as with an increasing percentage of MS from 4% to 12% but decreased at 16% percentage of MS.

![Figure 4. Compressive Strength of Mortar Specimens](image)

The compressive strength of the CM at the age of 3 days was 25.96 MPa. The mix MS1, MS2, and MS3 showed an increase of 5.97%, 9.17%, and 13.33%, but MS4 showed an increase of 12.10% as compared to CM. For MS3, the compressive strength was obtained as 29.42 MPa, maximum among all mixes. At 7 days of age, the compressive strength of the CM was 32 MPa. The mix MS1, MS2, and MS3 showed an increase of 7.72%, 11.56%, and 16.22%, but MS4 showed an increase of 13.53% as compared to CM. For MS3, the compressive force was measured as 37.19 MPa. At 28 days, the compressive strength of CM was 40.37 MPa. The mix MS1, MS2, and MS3 showed an increase of 10.45%, 15.06%, and 18.68%, but MS4 showed an increase of 17.17% as compared to CM. In this case, the maximum compressive strength was observed for MS3 as 47.91 MPa. At 56 days of curing age, the compressive strength of CM was 43.76 MPa. The mix MS1, MS2, MS3 showed an increase of 6.44%, 9.28%, and 15.04%, but MS4 showed an increase of 12.68% as compared to CM. The maximum compressive strength was observed for MS3 as 50.34 MPa. The comparative study reveals that the mortars mixes, with the replacement of cement by 10% GGBS and 12% MS exhibited increased compressive strength than the controlled mix, CM. This observation can be attributed to the pozzolanic activity of MS and GGBS [21]. Cement hydration creates calcium hydroxide and contributes to the formation of pores in the cement matrix. Silica particles in MS and GGBS react with calcium hydroxide to form calcium silicate hydrate gel that gets filled in the pores of the cement matrix. This increases the compressive strength of the cement mortar [10]. Besides, as the percentage of MS is increased in the cement matrix, it increases the pozzolanic effect of MS and improves its compressive strength. The sudden drop in compressive strength at a higher percentage of MS may be due to the reason that a higher percentage of MS can result in friction among particles leading to reduced packing and hence reduced compressibility [25]. Thus, 12% MS in presence of 10% GGBS is the optimal substituent dosage for the studied systems. The data was further comparatively analyzed in terms of age of curing days. An increase in compressive strength was observed in all the mortar mixes as evident in figure 5. The comparative account for percentage increase in compressive strength has been listed in Table 4. The data revealed that as compared to CM, the percentage increase of compressive strength in mortars with the replacement of cement with GGBS and MS was higher at increasing days of curing age up to 28 days. This observation confirms that the pozzolanic activity of
GGBS and MS strengthens the mortar mixes and hence increases the strength of such mixes as compared to CM. Similar trends have been reported by other researchers during their investigations [16,26].

Table 4. % Increase in Compressive Strength of Specimens

| Specimen | 3 Days | 7 Days | 28 days | 56 Days |
|----------|--------|--------|---------|---------|
| MS1      | 5.97   | 7.72   | 10.45   | 6.44    |
| MS2      | 9.17   | 11.56  | 15.06   | 9.28    |
| MS3      | 13.33  | 16.22  | 18.68   | 15.04   |
| MS4      | 12.10  | 13.53  | 17.17   | 12.68   |

**Figure 5.** Effect of Curing age on Compressive Strength of Mortar Specimens

3.3. **SEM Analysis**

The SEM analysis of all the mortar samples including CM, MS1, MS2, MS3, and MS4 were carried out for a comparative account of the difference in microstructure with partial substitution of cement by GGBS and MS. The microstructure of CM at 3 days of curing age exhibits that the structure is porous and many Ca(OH)$_2$ crystals are present (Figure 7a).
The structure is still porous after 7 days of curing age, with some cracks but comparatively lesser Ca(OH)$_2$ crystals are present (Figure 7b). Even after 28 days of curing, the cement matrix appears porous accounting for the low compressive strength (Figure 7c). The micrograph of MS1 at 3 days of curing shows some pores in the structure and calcium hydroxide crystals. Thus, the microstructure of MS1 with cement replaced by 4% MS in presence of 10% GGBS appears loose and that the calcium hydroxide has not been consumed much at the age of 3 days (Figure 8a). It shows that the pozzolanic reaction is still delayed which accounts for the only slight increase in strength as compared to that in the structure of CM [27]. The microstructure after 7 days shows comparatively fewer pores and also shows the calcium silicate hydrate crystals due to the commencement of the pozzolanic reaction (Figure 8b). However, the microstructure after 28 days appears comparatively denser and shows many calcium silicate hydrate crystals leading to high compressive strength as compared to CM (Figure 8c).

The microstructure of MS2 with cement replaced by 8% MS in presence of 10% GGBS is compact with lesser pores and that the calcium hydroxide has been quite consumed at the age of 3 days showing the initiation of the pozzolanic reaction accounting for the increased strength as compared to that in CM and MS1 (Figure 9a). With progression in curing age, the structure appears comparatively denser and shows many calcium silicate hydrate crystals leading to high compressive strength (Figure 9b and 9c).

The micrograph of MS3 (12% MS in presence of 10% GGBS) shows scanty pores in the structure and calcium hydroxide crystals as compared to that in the structure of CM, MS1, and MS2 (Figure 10). Thus, the microstructure of MS3 with cement replaced by 12% MS in presence of 10% GGBS is quite compact and that the calcium hydroxide has been quite consumed with progressing curing age...
showing the development of the pozzolanic reaction accounting for the increased strength [10]. The microstructures of MS4 are comparatively less homogeneous at the studied ages consistent with the strength analysis (Figure 11).

![Micrographs of MS3 at (a) 3 Days; (b) 7 Days; (c) 28 Days](image)

**Figure 9.** Micrographs of MS3 at (a) 3 Days; (b) 7 Days; (c) 28 Days

![Micrographs of MS4 at (a) 3 Days; (b) 7 Days; (c) 28 Days](image)

**Figure 10.** Micrographs of MS4 at (a) 3 Days; (b) 7 Days; (c) 28 Days

4. Conclusion

Based on the study, the following conclusions can be drawn: 1. Replacement of cement with GGBS and MS in presence of GGBS leads to an increase in consistency and delay of the setting process. This also increases the stiffness of the mortar and decrease of the flow. 2. The mixes with the replacement of cement by MS in presence of 10% GGBS showed an increasing order of compressive strength with maximum value at 12% MS. 3. The microstructure of mortar mixes without replacement of cement with MS and GGBS appeared more porous and less compact leading to lesser compressive strength. On contrary, the microstructure of mortar with 12% MS in presence of 10% GGBS appeared least porous and most compact with calcium silicate hydrate gel leading to higher compressive strength. Further, the combined cost of optimized content of GGBS and MS is comparable to that of cement and gives commendable strength enhancement. Thus, the partial substitution of cement by optimal amount of MS (12%) in presence of GGBS (10%) can lead to enhancement of compressive strength along with reduced cement use leading to lesser environmental hazards.

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