the use of blast furnace slag as a supplementary cementitious material

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Abstract: Waste material recycling in mortar production not only provides a promising resource for producing high-quality mortar, but it also aids in properly addressing the waste disposal issue. This study investigates the potential of utilizing blast furnace slag wastes as a supplementary cementitious material in blended cement-based mortars. The experimental program includes investigation of compressive strength of cement-based mortar composites contained different types and percentages of slag wastes. The mortar specimens were prepared and cured following related international standards. After curing, the compressive strengths of the tested specimens were assessed and compared with control specimens. The influence of the chemical composition of the investigated slag types on the resulted strength of the blended cement mortar was also discussed. Results of this study revealed that the chemical compositions of the raw slag wastes have significant effects on the pozzolanic activity of the produced slag. Slag samples contained higher percentages of calcium oxides present the most promising results regarding the production of supplementary cementitious material.

Keywords: blast furnace slag, Slag Activity Index, compressive strength.

1. Introduction:
Natural, waste, or by-product supplementary cementitious materials, have grown exponentially in the production of composite cements, for example, due to ecological, economic, and diverse product quality reasons, blast furnace slag has been used in the manufacture of cement since 1880. Slag or by-product of the transformation of iron ore into pig iron in a blast furnace is referred to as annual granulated blast furnace slag (GBFS). Production capacity in China is around 15 million tons [1, 2]. Its usage has grown since then as a result of its numerous advantages over other cementitious materials. To begin within comparison to fly ash, silica fume, and pozzolanas, slag has a relatively constant chemical composition, and other materials. It also has benefits such as low hydration heat, high sulfate and acid resistance, improved workability, and higher ultimate power, among others. Dams for hydroelectric power, massive energy, and bridges plants, metro systems, highways, and harbours benefit from these resources. Cement fineness is one of the most critical determinants of consistency. The increased fineness of the cement leads to a faster rehydration process as well as an increase in the speed of obtaining early compressive strength. Many researchers are interested in cement grinding, especially blended cement [3]
For large projects requiring a large amount of cement from earlier research work, reducing the required amount of Portland cement without compromising concrete performance is critical. Furthermore, the manufacture of Portland cement clinker requires a significant amount of energy. It has a significant environmental effect since it necessitates extensive quarrying for raw materials since it weighs 1.7 tons are needed to produce one ton of clinker and the release of greenhouse and other gases into the atmosphere. Per ton of clinker made, approximately 850 kg of CO₂ is emitted [4, 5]. As a result, pozzolans and cementitious materials play an essential role in the manufacture of concrete. Many organizations around the world have faced a complex problem with waste management in recent years. And businesses must figure out how to recycle their waste [6]. The use of waste in place of cement in concrete saves a lot of energy and has many environmental benefits. Furthermore, since cement accounts for more than 45 percent of the cost of concrete, it will have a significant impact on lowering concrete prices. According to some scholars, the best construction method use of waste from other sectors as construction materials is one way for the industry to become more sustainable [7, 8]. When finely ground and mixed with Portland cement (PC), such granulated slag has been found to have excellent cementitious properties. The reactivity of ground granulated blast furnace slag (GGBFS) is a key criterion for determining how efficient GGBFS is in concrete composites. To predict the hydraulic behavior of slag from a blast furnace, glass material, chemical composition, mineralogical composition, fineness, and the form of activation given influence slag reactivity. Pal, et. al. try to calculate the Hydraulic Index at 7 and 28 days by taking into account these critical parameters that influence slag characteristics [9]. Their findings revealed that concrete with a 30% replacement would achieve strong mechanical properties and durability. Using a grinder to enable the slag and clinker, the hydration and strength of cement are investigated. In Portland Slag Cement, mechanically activated granular blast furnace slag was used to replace the clinker (PSC). The findings indicate that an increase in reactivity causes a shift in microstructure and that slag densification is linked to an increase in cement strength. The fineness of slag is more important than the fineness of clinker in terms of strength growth [10, 11].

The aim of this study is to assess the effects of chemical composition of different types of blast furnace slag on the strength activity index of the produced slag to be utilized as a supplementary cementitious material with cement-based mortars.

2. Materials
Ordinary Portland cement, commercially known, (Lafarge), produced in Sulaymaniyah province was used in this study. To prevent exposure to weather conditions such as moisture, it was stored in a dry location. The cement's chemical composition and physical properties in this project are shown in Tables 1 and 2, respectively. The results show that the cement meets Iraqi requirements [12].

Table 1: Physical properties of cement.

| Physical properties | Test results | Limits of I.Q.S No. 5/1984 |
|---------------------|--------------|--------------------------|
| Specific Surface area m²/kg | 293 | > 230 |
| Soundness by Autoclave | 0.19 % | < 0.8 % |
| Setting time, hrs. : min. | 2:35 | > 00:75 |
| Initial setting time. | 3:60 | < 10:00 |
| Final setting time. | | |
| Compressive strength | | |
| 3-day MPa | 18.5 | > 15 |
| 7-day MPa | 25.3 | > 23 |

* Physical test were carried out by the National Center for Geological Survey and Mines.
Table 2: Cement’s chemical structure and core constituents.

| Composition of Oxide | Abbreviation | Content % | Limits of (IQS NO.5/1984) |
|----------------------|--------------|-----------|---------------------------|
| Lime                 | CaO          | 60.91     | -                         |
| Silica               | SiO2         | 20.25     | -                         |
| Alumina              | Al2O3        | 4.67      | -                         |
| Iron oxide           | Fe2O3        | 3.92      | -                         |
| Sulfate              | SO3          | 2.23      | < 2.8 % If C3A > 5%       |
| Magnesia             | MgO          | 3.22      | <= 5%                     |
| Loss on Ignition     | L.O.I        | 2.56      | <= 4%                     |
| Insoluble Residue    | L.R          | 0.9       | <= 1.5%                   |
| Lime Saturated Factor| L.S.F        | 0.93      | 0.66-1.02                 |

Name of Compound     | Formula               | Abbreviation | Percentage by Weight |
Tricalcium silicate  | 3 CaO.SiO2            | C3S          | 55.673                 |
Dicalcium silicate   | 2 CaO.SiO2            | C2S          | 27.125                 |
Tricalcium silicate  | 3 CaO.Al2O3           | C3A          | 7.122                  |
Tetracalcium         | 4 CaO.Al2O3.Fe2O3     | C4AF         | 9.559                  |

* Chemical test were carried out by the National Center for Geological Survey and Mines.

2.2: Sand
Standard silica sand was selected as a fine compound, consisting almost entirely of naturally round grains of semi-pure quartz, used to prepare the cement-based mortars. Its classification and physical properties are explained in Tables 3, 4 and 5. All of them fulfill the requirements of (ASTMC778, 2013) [13], [14, 15], respectively.

Table 3: The requirements of (ASTM C778, 2013)

| Sieve size(mm) | Passing by weight | Limit of Passing percentage according to ASTM Specification % |
|----------------|-------------------|-------------------------------------------------------------|
| 1.18 mm (No. 16)| 100               | 100                                                         |
| 850 μm (No. 20) | 99.5              | 85 to 100                                                   |
| 600 μm (No. 30) | 4                 | 0 to 5                                                      |

Table 4: Iraqi specification (IQS No.2080/1998)

| Sieve size(mm) | Passing by weight | Limit of Passing percentage according to IQS No.2080/1998 Specification % |
|----------------|-------------------|--------------------------------------------------------------------------|
| 850 μm (No. 20) | 99.5              | 98 min                                                                   |
| 600 μm (No. 30) | 4                 | 10 max                                                                   |

* The National Center for Geological Survey and Mines carried out the tests.

Table 5: The characteristics of the sand used in this project.

| Physical properties | Test result | Limit of Iraqi specification No.45/1984 |
|---------------------|-------------|-----------------------------------------|
| Specific gravity    | 2.66        | -                                       |
Absorption %  0.96
Sulfate content %  0.37  0.5%

* Test results were carried out by the National Center for Geological Survey and Mines.

2.4 Additives:
Master Glenium 51 has been primarily developed for applications in the ready mixed and precast concrete industries where the highest durability and performance is required is free from chlorides and complies with ASTM C494 Types A and F, compatible with all Portland cements that meet recognised international standards [16]. Super plasticizers of the past, Sulphonated melamine and naphthalene formaldehyde condensates, for example, are absorbed onto the surface of the cement particles during mixing. Table (6) as shown below shows the chemical characteristics of this additive.

| Table 6 characteristics of chemicals admixture |
|-----------------------------------------------|
| Appearance                        | Viscous Liquid                  |
| Relative density                  | 1.1± 0.005kg/L                  |
| Chloride content                  | Nil                             |
| Viscosity                         | 128 +/- 30 cps @ 20°C           |
| PH value                          | 6.6                             |
| Transport                         | Not classified as dangerous     |
| Colour                            | Light Brown                     |

Blast-furnace slag is a non-metallic material composed primarily of calcium and other bases' silicates and aluminosilicates; granulated slag is the glassy or noncrystalline product formed when molten blast-furnace slag is quickly cooled, such as by immersion in water. Type I cement is made up of a near and consistent blend of Portland cement (or Portland blast-furnace slag cement) and fine pozzolan, with the pozzolan content ranging from 15 to 40% of the total cement mass. A pozzolan is a siliceous or siliceous and aluminous substance that has no intrinsic value or no cementing property, but will chemically react with calcium hydroxide in finely divided form and in the presence of moisture at ordinary temperatures to form cementitious compounds. Finely ground granulated blast-furnace slag is self-cementing compared to pozzolans, meaning it requires calcium hydroxide to form cementitious products like C-S-H. [17]

2.5 Characteristics of GGBFS in terms of both physical and chemical properties
2.5.1: physical and chemical Characteristics:
The slag has a specific gravity of about 2.90 and a bulk density of 1200–1300 kg/m3. The color of GGBS is a light gray or white powder with little immediate risk. A single, brief encounter with that material is unlikely to result in serious injury. Table (7) as shown below describes the physical and chemical characteristics of GGBFS. Exposure to the wet material, on the other hand, may result in extreme, potentially irreversible tissue damage. Chemical (caustic) burns cause (eye) damage. Chemical (caustic) burns or an allegoric reaction may cause tissue damage in wet or damp areas of the body if they are exposed to the substance for long enough. Use the MSDS-recommended exposure controls or personal safety methods.
Table 7 Physical and chemical characteristics of GGBFS. (From Producer Data Sheet)

| Properties                                      | Standard Requirements | Values       |
|-------------------------------------------------|-----------------------|--------------|
| MgO                                             | ≤18%                  | 5.00 - 10.00 |
| Sulfide                                         | ≤2.0 %                | 0.40 - 0.70  |
| Sulfate                                         | ≤2.5 %                | 0.30 - 0.60  |
| Loss on ignition, corrected for oxidation of sulfide | ≤3.0 %              | 0.00 - 2.00  |
| Chloride                                        | ≤0.10 %               | 0.01 – 0.03  |
| Moisture content                                | ≤1.0 %                | 0.10 – 0.30  |
| Specific surface (Blaine) cm²/g²                 | ≤2750                 | 5800 - 6100  |
| Activity index at 7 days                        | ≤45 %                 | 50-60        |
| Activity index 28 days                          | ≤70 %                 | 75-85        |
| Glass content (%)                               | 67 (min.)             | 90           |
| Chemical moduli (a) CaO+MgO+SiO₂                 | 70 (min.)             | 82.72        |

2.6: Experimental Work
The Experimental work of this study was carried out as indicated in the points below
1. The first stage entails choosing suitable materials and determining their proportions to be partially replaced with cement particles.
2. Mixing, casting, and curing mortar specimens are all part of the second step.
3. New, hardened, and microstructure properties are tested in three stages
4. Compressive two ages 7 and 28 days are included in the fourth level, which involves assessing mechanical and microstructure characteristics.

2.6.1 Slag Activity Index test:
ASTM C989 defines SAI as the percentage ratio of the average compressive strength of slag cement (50% -50%) mortar cubes to the average compressive strength of reference cement mortar cubes at a designated age, expressed as: Slag Activity Index, percent SAI = (SP/Px100) where
SP = average compressive strength of slag-reference cement mortar cubes [psi];
P= average compressive strength of reference cement mortar cubes [psi]. Based on this, slag was classified into three grades— Grade 80, Grade 100 and Grade 120 depending upon their respective compressive strength. Classification is in accordance with Table 8 (ASTM C989).
Figure 1 shown below shows the method for casting the cubes for testing Slag Activity Index.

Table 8 SAI standards for various grades as prescribed in ASTM C989

| Age and grade | SAI minimum percent |
|---------------|---------------------|
|               | Average of last five consecutive samples | Any individual sample |
| 7-day index   |                     |                     |
| Grade 80      | –                   | –                   |
| Grade 100     | 75                  | 70                  |
| Grade 120     | 95                  | 90                  |
| 28-day index  |                     |                     |
| Grade 80      | 75                  | 70                  |
| Grade 100     | 95                  | 90                  |
| Grade 120     | 115                 | 110                 |
2.6.2 Mixing, casting and curing

Mixing process that was followed during the work program in accordance with ASTM C305-14 [18] as following:

- Super plasticizer should be well dispersed in water, applying the mix water to the container, and then applying the cementitious materials which include cement and blast furnace slag, all mixture should be blended at lowest speed during 30 seconds.
- Adding standard sand, firstly, the mixing should be at 30 seconds at the lowest speed, then medium speed is selected for mixing during the same period.
- 90 seconds period must be left without mixing, scrapping the mixture that was remaining on the blades and bowl walls throughout the first 15 seconds.
- Blending the mixture at medium speed for 1 min.

Table (9) provide details for each combination, including mix proportions and ingredients. Trial mixes were created to reach a flow rate of (110±5) percent at a constant water/binder ratio. ASTM C1437-13 specifies the use of variable percentages of super plasticizer. As can be seen in Figure 2 cubic molds of (50x50x50 mm) were selected to all mixtures types to be casted into, and should be vibrated for 30 s. to remove air voids. After 24 hours of casting, molds should be removed as displayed in figure (1) and (2), and then the specimens were cured in clean water for 7 and 28 days as shown in figure (3) and (4).

Table 9: Mixture proportion of mortar

| code of specimens   | w/b | Water | Cement | Sand  | Blast furnace slag | HRWR |
|---------------------|-----|-------|--------|-------|--------------------|------|
| Control             | 0.485 | 242   | 500    | 1375  | 0                  | 3    |
| Blast furnace slag(A) | 0.485 | 242   | 250    | 1375  | 250                | 3    |
| Blast furnace slag(B) | 0.485 | 242   | 250    | 1375  | 250                | 3    |
2.6.3 Fresh Mortar Tests
2.6.3.1 Flow test
Flow test is performed to describe the workability property of mortar mixtures which is considered one of the most essential characteristics for mixture evaluation. The flow test method is carried out in accordance with ASTM C1437-13 [19] including the following:

\[
\% \text{ Flow} = \frac{100 - \left(\text{average of 4 readings of diameter}\right)}{100} \times 100
\]

2.6.3.2 Hardened mortar Test
Mechanical experiments were used in this analysis to evaluate hardened blended cement mortar containing blast furnace slag. (compressive strength)

2.6.3.2.1 Compressive strength
This test was carried out on cubic specimens with dimensions of 50mm x 50mm x 50mm, with the load applied vertically at the cast surface in compliance with ASTM C109/C 109M-
As shown in Figure 4, the test was performed using ELE International's digital concrete compression equipment at a loading rate of 1,500 kN/min. This system also provided the load and compressive strength in kN and Mpa, respectively. The following equation predicted the compressive strength:

\[ F_m = \frac{P}{A} \]

Where:
- \( P \): total ultimate load in kN
- \( A \): loaded surface's area in mm\(^2\)

This system also provided the load and compressive strength in kN and Mpa, respectively. The following equation predicted the compressive strength:

The effect of varying Portland cement concentrations replacement with blast-furnace slag on the hardened concrete's resistance to real compression \((f_c)\) at 7 and 28 days is worth noting. The cementing material mixtures that have been studied are made up of the following system: Slag and Portland cement. The volume of water used in the concrete mixture preparation is usually implied indirectly in the water/cement relationship.

Curing methods for concrete after 24 hours of strain include controlled relative humidity curing, humid curing or water immersion curing, room temperature curing, temperature \((T)\) curing \((HR)\). The cementing process used for the concrete mixture with a slag dosage as a cement substitute can decide the curing period, as shown in Table 2. While there are several options for using BFS as a partial substitute for cement in the manufacture of concrete mixtures, the outcome may not always be favourable in terms of mechanical resistance. As a result, some information about the concrete's compression resistance in comparison to the slag used in the OPC-BFS cementing method must be given [17].

3.7 Microstructure tests
3.7.1 X-ray diffraction spectroscopy (XRD)
X-ray diffraction (XRD) is a multilateral, non-destructive technique for detecting the crystallographic structure of natural and artificial materials for mineral admixtures (blast furnace slag), implying specifics and knowledge about broken surfaces; (XRD) is a reasonably cost-effective method for detecting and counting cement mineralogy. (Raikar, 2012).

Any crystalline compound has a unique peak property that can be identified by its location and intensity. When a mineral's crystalline is exposed to an X-ray of a certain wave length, layers of atoms deviate rays and produce peaks that reflect the mineral's properties. The amplitude of unique peaks from different individual minerals was proportional to the volume of the specimen that contained more than one mineral when Xrayed. Nanotechnology and advanced materials research center/university of Technology conducted XRD tests for this study, as shown in Figure 5, 40 kV voltages and 30 A current were measured using an X-ray diffract meter. Diffraction scans over a 2 range of temperatures can reveal the minerals phases.
(10-90). After cooling to room temperature, about 90 gm powder was taken from the inner part of the specimen and dried at 100 °C to perform the XRD test.

Figure 5 XRD

3.8: Results and Discussion
3.8.1 Compressive strength
The compressive strength test was conducted using cubes (50mm). The cubes tested by using (ELE) machine and an average of three cubes was recorded. The age of tests was 7 and 28 days and presented in Table (10) and Figs. (6,7). As can be seen, the compressive strength of specimens containing both types of slags A and B reduced at 7 days compared to control. However, the reduction rate was more pronounced in slag type B. The 7 days reduction rates were 8% and 45% in specimens modified with type A and Type B respectively, compared to control specimens. In contrast, at 28 days age type A slag showed enhanced strength properties as the compressive strength recorded 8% improvement than control specimens. Fig. 7 also shows that mortars modified with type B slag exhibited reduced compressive strength even in 28 days age. This superiority of Product A could be attributed to the higher pozzolanic activity of its particles due to higher calcium oxide contain revealed by the chemical analysis conducted during the preparation stage. These results suggest that slag type A overcome slag type B at both early and later ages (i.e. up to 28 days), and therefore became the preference for use as a supplementary cementitious materials with cement-based mortars.

Table 10 Compressive strength test of mortar

| Mixture                        | Compressive strength |
|--------------------------------|----------------------|
|                                | 7day                | 28day                |
| Control (average)              | 9.7                 | 21.2                 |
| Blast furnace slag(A)(average) | 7.9                 | 22.9                 |
| Blast furnace slag(B)(average) | 5                   | 10.1                 |
Figure 6: Compressive strength at 7, 28 days

Figure 7: Change % of Compressive Strength at 7 and 28 days

3.8.2 X-ray diffraction spectroscopy (XRD)

Table (12) and Figures 8 and 9 show blast furnace slag chemical structure and core constituents type A and B respectively.

| Element        | Symbol | Concentration (A) | Concentration (B) |
|----------------|--------|-------------------|-------------------|
| Sodium         | Na2O   | 0.802%            | 0.228%            |
| Magnesium      | MgO    | 6.149%            | 0.5765%           |
| Aluminum       | Al2O3  | 10.31%            | 4.532%            |
| Silicon        | SiO2   | 37.20%            | 69.98%            |
| Phosphorus     | P2O5   | 0.0371%           | 0.0243%           |
| Sulfur         | SO3    | 1.551%            | 0.2133%           |
| Chlorine       | Cl     | 0.02002%          | 0.08737%          |
| Potassium      | K2O    | 0.7871%           | 0.2336%           |
| Calcium        | CaO    | 39.37%            | 6.752%            |
| Titanium       | TiO2   | 1.071%            | 2.180%            |
| Vanadium       | V2O5   | < 0.00018%        | 0.0528%           |
| Chromium       | Cr2O3  | 0.00331%          | 2.653%            |
| Manganese      | MnO    | 2.612%            | 13.85%            |
| Iron           | Fe2O3  | 0.9223%           | 5.390%            |

Table 12: Blast furnace slag chemical structure and core constituents type A and B.
The chemical analysis shown in Table revealed that the concentrations of SiO\textsubscript{2} were 37.2\% and 69.98\% for slags type A and type B respectively. The CaO concentrations were 39.3\% and 6.75\% for Types A and B respectively. The superior pozzolanic activity of slag type A is seemed to be correlated to the higher concentration of CaO compounds, while the SiO\textsubscript{2} concentrations were played insignificant role in the slag activity when added to cement-based materials. This behavior confirm that the CaO contained was the dominant factor in increasing its pozzolanic activity of blast furnace slag waste materials.

8. Conclusions:

Based on the results of this study, the following conclusions can be made:

1- Through the process of conducting a compression test for cement mortar samples containing cement (a control mixture) as well as samples containing cement and blast furnace slag in both types A and B it was found that samples containing blast furnace slag type A are better than type B. This is because pozzolanic materials have slow reactions at early ages and do not give high compressive strength, as for the increase in the compressive strength of the blast furnace slag type A at the age of 28 days.

2- The superior pozzolanic activity of slag type A is seemed to be related to the higher concentration of CaO compounds. This behavior confirm that SiO\textsubscript{2} concentrations have insignificant role in the slag activity when added to cement-based materials, while the CaO contained was the dominant.

3- Through the process of mixing, cast, and compression testing of the samples, the results showed that the color of mortar containing slag is lighter than normal Portland-cement.

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