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Impact of Waste Iron Slag on Mechanical and Durability Properties of Concrete

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ABSTRACT

Waste management is of great concern in today's world. Every year, an enormous amount of solid waste is generated from different industrial activities, especially the waste which is produced by iron industries in a particular form of slag. The major issue of emission of carbon dioxide (CO_2) from cement industries is a serious problem for the earth's environment and the surrounding area. Thus, in this study, the waste iron slag obtained from nearby iron industries was used as a partial substitute for cement. The cement was replaced with iron slag (IS) at the substitution levels of 7.5%, 15%, 22.5%, 30% and 37.5% by weight of cement. The doses of superplasticizer for every mix were taken based on the essential workability requirements for the reinforced-concrete work. Performance of control and blended mixes was evaluated by workability evaluation, compressive strength test, flexural strength test, water permeability test, water absorption evaluation, rapid chloride penetration test (RCPT) and carbonation test. Scanning electron microscopy (SEM), X-ray diffraction (XRD) technique and thermogravimetric analysis (TGA) technique were used to assess the micro-structural changes and to evaluate the chemistry of the blended mixes. The results obtained from this study were encouraging in terms of compressive and flexural strengths. The maximum compressive and flexural strengths were recorded at a 22.5% replacement level of slag. The results obtained at 30% replacement were also better compared to the control mix. The resistance of slag-made concrete mixes against adverse conditions; i.e., CO_2 penetration, chloride penetration and water penetration was far better than that of conventional ones. The results obtained from TGA indicated that the productivity of calcium silicate gel of slag concrete is better than that of control concrete.

KEYWORDS: Concrete, Slag, Carbonization, RCPT, XRD, TGA, SEM.

INTRODUCTION

Concrete has been widely used for the betterment of infrastructure, which helps improve the quality of life of common people. In the construction industry, around 400-500 million metric tonnes of cement are required every year to produce concrete (Dhanesh et al., 2019). Cement and concrete industries are harming the environment and creating problems related to health issues, among others. The concrete and cement industries are among the major sources of CO_2 emission (Jain et al., 2022). Different types of admixtures are being used in concrete industries for the invention of sustainable concrete. Mineral admixtures (fly ash, rice husk ash, silica fume, … etc.) are also being used to produce robust concrete. In the current scenario, many supplementary cementitious materials (SCMs) are preferred to resolve the issues of the environment and the second thing is to reduce the production of cement (Alizadeh et al., 2003). The manufacturing industries of concrete produce around 800 billion tonnes of concrete

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every year from different plants of concrete industries. The production of waste iron slag from iron industries creates a lot of problems for nearby areas, especially health and environmental issues (El-Didamony and Amer, 2016). Slag, when finely ground, has been found to exhibit excellent cementitious properties. Used with Portland cement, slag is a byproduct acquired in the production of pig iron in the blast furnace and its silicate content and alumino-silicates of calcium and of other bases provide concrete with better strength (Ozbay et al., 2016). Maximum packing density and compressive strength were achieved at 17% replacement of cement with slag. Micro-structural studies have reported the formation of C-S-H gel with densification and less Ca/Si ratio when compared with the concrete control samples (Krishna and Kumar, 2020). Nadeem et al. reported the production of concrete by using slag and fly ash as cement-replacing materials to have enhanced the mechanical and durability performances of concrete. Maximum compressive and flexural strengths were achieved at 10% and 20% replacement of cement with fly ash and slag. Dense filling and less porosity were the suitable reasons of improving compressive and flexural strengths (Nadeem and Pofale, 2012). The use of slag as partial replacement of cement in % of 3, 5, 7, 10, 13, 15 & 18 by weight of cement increased the properties of concrete in terms of permeability and water absorption. The use of 15% blast furnace slag (BFS) reduced water permeability and water absorption of blended mixes (Gururaj et al., 2015; Pade and Guimaraes, 2007). Prepared concrete with slag and nano-silica improved the long-term behavior of concrete. The highest compressive strength was obtained at 20% replacement and was 13.36% greater than that of the control specimen (Abukersh and Fairfield, 2011; Sancheti et al., 2020). Naik et al. analyzed the effect on self-compacting concrete by using iron slag as a powder in concrete. Durability and mechanical properties were improved with an increase in the amount of blast furnace slag (Gautam et al., 2014). Partial replacement of fine aggregate by fly ash and cement by slag in concrete led to an improvement in micro-structural properties of concrete. Maximum results were obtained at 30% replacement of these dual-character materials (Nakum et al., 2015). The incorporation of slag as a partial replacement of cement in concrete improved the behavior of concrete against adverse effects (Harison et al., 2014). In high-performance concrete, the use of slag as a partial replacement of cement by weight improved compressive and flexural strengths. Maximum strength was achieved at 20% replacement with slag. The results of the RCPT test indicated that an increase in slag reduces the permeability with a reduction of charge (Nazari and Riahi, 2011). Addition and replacement of slag in concrete improved the durability properties and reduced the chloride diffusion (Rahman, 2021). The reported use of slag with fiber improved the flexural performance of concrete in terms of flexural strength. Further, durability performance of concrete-like acid improved against sulphate attack (Saurav and Kumar Gupta, 2014).

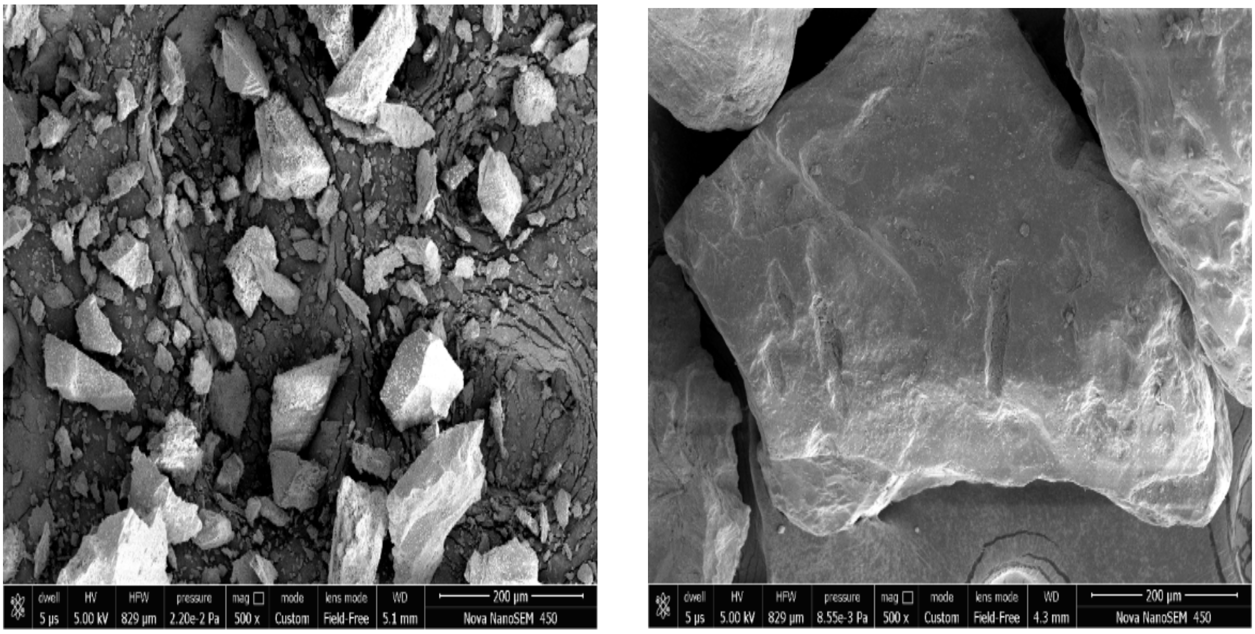
From the literature, it was found that slag as a binder improved the properties of conventional concrete. The waste slag is available in huge amounts all over India, especially in the western and southern parts of the country. The main objective of this study was to evaluate enhancing the performance of concrete by using waste slag to improve the performance of conventional concrete. The other objective was to obtain a solution for the problem of CO_2 emissions in the environment due to the production of cement from industries. Water permeability, water absorption, rapid chloride penetration, carbonization, compressive strength and flexural strength were tested and analyzed in this study. SEM and XRD techniques were also used for the analysis of micro-structural changes.

MATERIALS AND METHODS

Materials

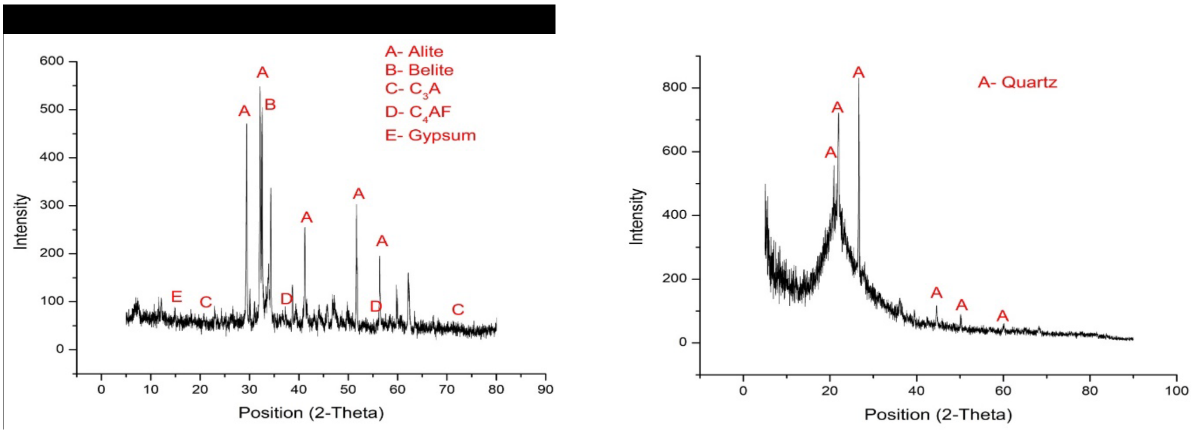
Ordinary Portland cement (OPC) of grade 43 (IS: 8112, 2013) was preferred for preparing the control concrete as well as the blended mixes containg different amounts of iron slag. Belongings of OPC cement are specified in Table 1. For fine aggregate (FA), river sand was used after sieving with a 4.75-mm sieve as prescribed in IS code (383, 1970). Zone- II of river sand was identified after sieve analysis as per IS 383. Coarse aggregate (CA) in the sizes of 20 mm and 10 mm was used for all mixes. Coarse aggregate was acquired from the nearby area of Jaipur, Rajasthan, India. Properties of coarse and fine aggregates are mentioned in Table 1. Cement and slag were used after sieving with a 90-micron sieve. Slag was collected from a local vendor in

Jaipur, Rajasthan, India. The physical and chemical properties of cement and slag are presented in Table 1 and Table 2, respectively. The particle-size distribution curves for coarse aggregate and fine aggregate are given in Fig.3. The plasticizer used in this study was neptha-based and obtained from a nearby vendor in Jaipur, Rajasthan, India. The plasticizer was used in 1%, 1.2%, 1.4%, 1.5%, 1.6% and 1.7% by mass of cement for control, 7.5%, 15%, 22.5%, 30% and 37.5% mixes, respectively. The SEM images and XRD patterns of cement and slag are shown in Fig.1 & Fig. 2.



(a) Cement **(b) Iron slag**

Figure (1): SEM images of cement and slag



(a) OPC-43 grade **(b) Iron slag**

Figure (2): XRD patterns of cement and slag

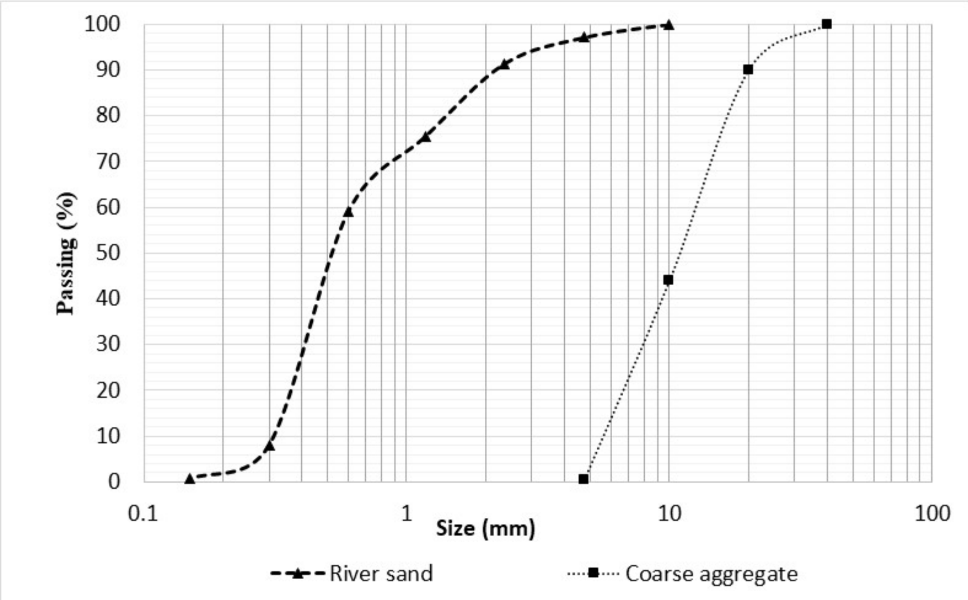


Figure (3): Particle-size distribution curves of river sand and coarse aggregate

Table 1. Properties of cement, aggregate and slag

Material	Water Absorption (%)	Specific Gravity	Bulk Density (kg/m^3)	Fineness Modulus (%)	Initial Setting Time (min.)	Final Setting Time (min.)
Cement	-	3.14	1445	2.47	40	145
FA	1.25	2.63	1578	3.10	-	-
CA	0.98	2.68	1589	7.25	-	-
Slag	1.26	2.92	1590	0.85	-	-

Table 2. Chemical compositions of cement and slag

Oxide	Cement (%)	Slag (%)
CaO	64	32
SiO_2	23	59
Al_2O_3	4	20
Fe_2O_3	3	2.5
MgO	1.5	5
SO_3	1.5	2
Na_2O	0.4	-
Gypsum ($CaSO_4.2H_2O$)	2.6%	-

Mix Design Procedure

Mix design was conducted as per the specifications given in the Indian standard code (IS 10262, 2019). Control and blended concrete mixes were checked on different performance parameters as per the pre-decided objectives. The casting of various specimens was done in cubes and beams of various sizes. The specific gravity of cement and slag was 3.14 and 2.92, respectively. The mix proportions are depicted in Table 3 for control and blended concrete mixes.

Table 3. Mix design of slag concrete (In 1m^3)

S.No.	Mix Name	Cement (kg)	Slag (kg)	Coarse Aggregate (kg)		FA(kg)	Water (kg)	Admixture (kg)
				20 mm	10 mm			
1	Control	410	0	710	474	680	168	4.1
2	IS 5	390	21	710	474	680	168	4.92
3	IS10	369	41	710	474	680	168	5.74
4	IS 15	349	62	710	474	680	168	6.15
5	IS20	328	82	710	474	680	168	6.56
6	IS25	308	103	710	474	680	168	6.97

The experimental work is carried out by casting two hundred and four cubes in size of 100×100×100 mm for compressive strength, carbonation attack and water absorption. For flexural-strength test, forty-eight beams were cast in size of 500×100 mm. Cylinders of 50-mm height and 100-mm diameter were cast for rapid chloride-penetration test.

Slump Test Procedure

Workability test of slump was performed as per the guidelines mentioned in (IS 1199, 1959). The apparatus of cone shape was filled with concrete in four layers. Each layer of concrete was filled in equal height. Each part of the layer was tapped 25 times with the help of a steel rod. Compaction of concrete was kept uniform throughout tempering. This procedure was repeated three times for each concrete mix. The difference between the height of the apparatus and the highest point of concrete is known as the slump value. The slump value in mm was determined with the help of a gauging scale. Slump Value = H-H^1

where H= Height of specimen or apparatus;
H^1 = Height of the highest point of material.

Compressive Strength Test Procedure

This test was generally commenced for the calculation of the strength of the concrete cubes. The Indian standard code used for calculating the compressive strength was (IS 516 -1959). The size of the cubical specimens used to evaluate compressive strength was 150 mm and 100 mm. After casting, all prepared samples were kept in a curing tank for 7,14,28,56 and 90 days. After exposure to water, the specimens were removed from the tank and their surfaces were cleaned with the help of a dry cloth. Immediately after that, specimens were placed between the plates of the compressive testing machine. The rate of loading was maintained at 140 kg/cm^2/min. The load was applied in the vertical direction to the specimen. The procedure was repeated thrice for each control and blended concrete sample. For the calculation of compressive strength, the expression is shown in Eq. 1.

$$\text{Compressive strength in (MPa)} = \frac{P}{A} \quad (1)$$

where P = Load (N);
A = Area of the specimen (mm^2).

Flexural Strength Test Procedure

Beam specimens of dimensions 100 × 100 × 500 mm were cast for determining the bending strength. Cast molds were kept in a rectangular water tank for up to 90 days at room temperature. The code referred to for performing the flexural-strength test was (IS 516, 1959). Four-point loading setup was used with two steel rollers placed at 1/3 distance from the ends. The load was applied gradually *via* rollers at the middle of the specimen. Load was increased until the specimen failed and the maximum bending strength value was noted down. For the calculation of flexural strength, the expression is shown in Eq. 2.

$$\text{Flexural strength of sample} = \frac{PL}{bd^2} \quad (2)$$

where
P = Load (N);
L = Length of the beam (mm);
b = Width of the beam (mm);
d = Depth of the beam (mm).

Water Absorption Test Procedure

The water-absorption test was accomplished as per the guidelines of (ASTM C 642). In this experimental work, 100-mm cube size specimens were cast and kept in the curing tank for a period of 28 days. After that, the specimens were kept in the oven for 24 hours to dry. The weight of oven-dried specimens was recorded and the specimens were placed again in the water bath for two days of curing. After two days, the specimens were removed from the curing tank and weighed. Water-absorption assessment was distinguished in terms of the ratio of the fully saturated sample to that of the oven-dried sample.

Water Permeability Test Procedure

In this test, the reference code was (DIN 1048, 1991). The specimen size was prepared to be 150 mm in cubical shape. The specimens were kept in a curing tank for 28 days and then removed from the water tank. After weeping the surfaces of the specimen, they were placed on the permeability test apparatus for 72 hours under a constant pressure of 15kg/cm^2 applied to the specimens' top surfaces. After 72 hours, the specimens were removed from the machine and the depth of water penetration was recorded by breaking the specimens into two halves using the compressive testing machine. The depth of penetration was noted in millimeters (mm).

Rapid Chloride Penetration Test (RCPT)

For this test, (ASTM C1202, 2012) was referred to in order to find the value of charges in coulombs (C). The size of the specimens cast was 100 mm in cubical shape and the specimens were kept in a curing tank for 28 days. The specimens were placed on the RCPT machine and the flow of charges through the samples. In this procedure, two chemical solutions were used, in which one was an NaOH solution and the other one was an NaCl solution. The whole procedure was conducted for 600 minutes and the readings were taken at 30-minute intervals. The charge noted in Coulombs (C) indicated the permeability as low, moderate or high.

Carbonation Test Procedure

The provisions of RILEM CPC 18 (ASTM C 267, 1998) were used for performing the test. The beam specimens of 50 × 50 × 100 mm size were cast to calculate the resistance against CO_2 attack. The specimens were protected with epoxy paint from all four sides except the longitudinal one and kept in a carbonation chamber. CO_2 concentration, humidity and temperature were maintained throughout the test duration. The test specimens were split longitudinally and a solution of phenolphthalein indicator was sprayed over the specimens at the end of each exposure period. The depth of the colorless area was recorded.

Micro-structural Analyses

SEM Analysis: A scanning electron microscope was used for the analysis of control and iron slag specimens. In this test, 10-mm size was used for control and slag concrete in the SEM analysis. This analysis is generally used for identifying the different phases and compounds in the concrete. The test was conducted at the Material Research Centre (MRC, Jaipur).

XRD Analysis: In XRD technique, mineral composition was analyzed for control and blended mixes with the help of an XRD machine. For performing this analysis, powder form of control and iron slag specimens was used. The samples were kept on a fiber-glass plate and an XRD machine was used for analysis. After analyzing the data, a curve is plotted between intensity and angle.

TGA Analysis: TGA was checked on the fine powders using a Q1000DSC+TGA instrument. The parameters involved were as follows: test temperature range: room temperature (RT) to 900°C; heating rate: 10°C/min; atmosphere: N2; test reference material: Al_2O_3. In the current study, the results received from TGA analysis were used to assess the CH content in the blended mortar from the mass loss measurement.

RESULTS AND DISCUSSION

Slump Test Results

The slump value with the incorporation of slag is decreased, as shown in Fig. 4. However, the minimum slump was achieved for each mix as per the requirements of RCC work. The angular and rough texture of slag (presented in the SEM image shown in Fig. 2) was a practical reason for reducing the slump value. In similar studies, authors also reported irregular and rough textures of slag particle corners tending to the interface with each other and due to this, friction is introduced and reduces the workability (John and John, 2013). Another reason was that the water pre-occupation capacity of slag is high compared to that of cement (Ipieca et al., 2015).

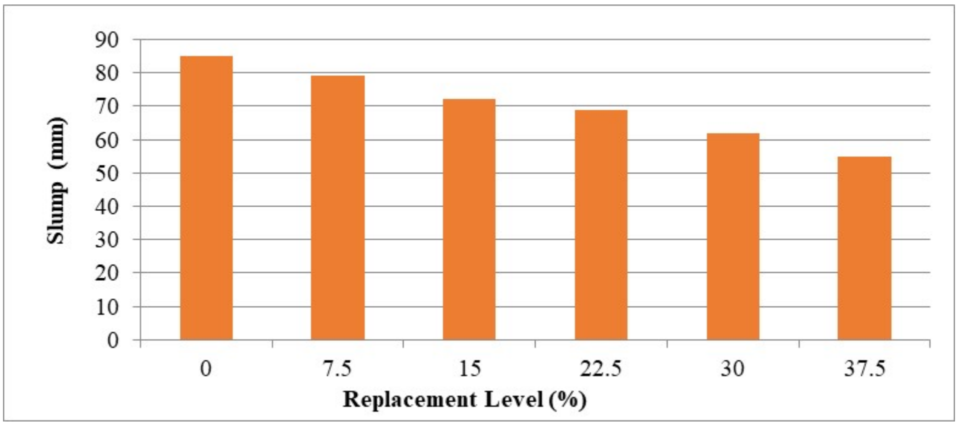


Figure (4): Slump-test results

Compressive-strength Test Results

Compressive-strength results for 7, 28, 56 and 90 days are shown in Fig. 5. Compressive strength improved with the incorporation of optimum 22.5% of slag-made concrete. The reason was the reduction in voids, which shows a compact and dense structure packing (shown in SEM image in Fig. 10). Some other authors have evaluated the strength development of slag-mixed concrete and reported that up to 40% of slag resulted in increased compressive strength. Slag-to-cement ratio played an important role in determining the efficiency of the mix (Harison et al., 2014). The reported higher surface area of slag requires additional water for absorption and it has reduced the water-to-binder ratio (w/b) and hence improved the strength (Saurav and Kumar Gupta, 2014). A higher percentage of slag-added concrete reduced the compressive strength for the concrete mixes. The reasons were voids and less packing density at higher replacement levels. The same has been reported by Cordeiro et al. (2016).

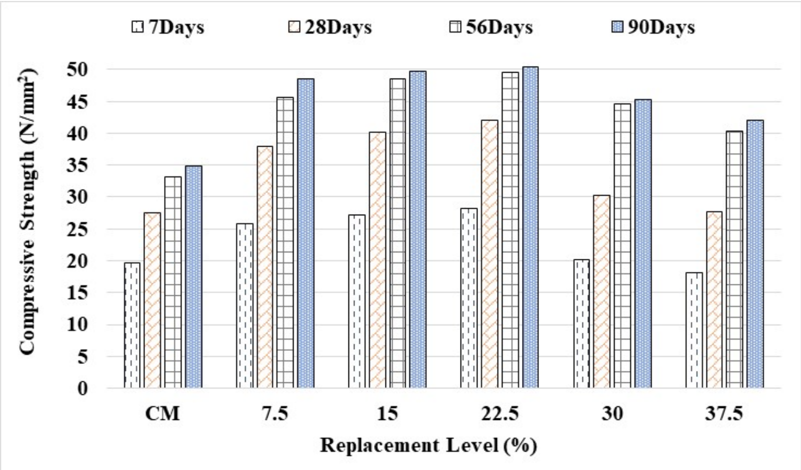


Figure (5): Compressive-strength test results

Flexural-strength Test Results

Flexural strength at 28, 56 and 90 days with the optimum percentage of slag-made concrete is presented in Fig. 6. The flexural-strength value increased with incorporating the optimum percentage of slag in concrete. The reason for improved strength was the pozzolanic behavior of slag, which converted additional calcium hydroxide (CH) into calcium silicate hydrate (CSH) gel (see XRD graph in Fig. 12). A higher proportion of slag fines in concrete mixes reduced the flexural strength. The reason was a reduction in the amount of crystalline $Ca(OH)_2$ that stopped the development of CSH gel (John and John, 2013).

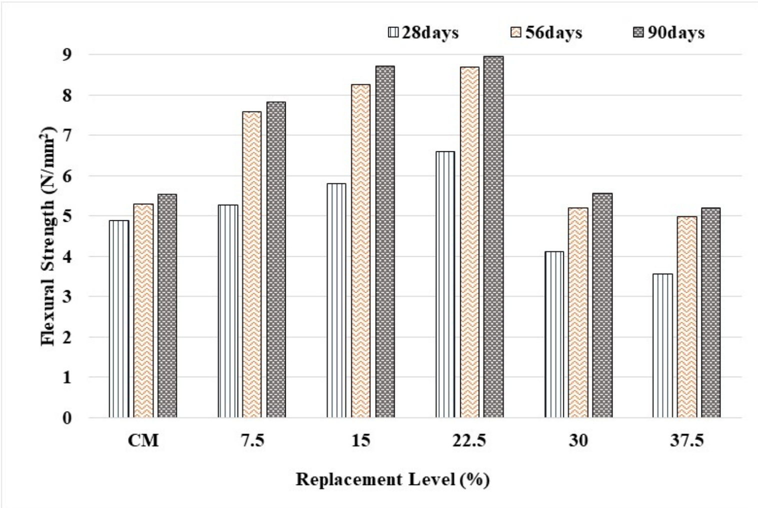


Figure (6): Flexural-strength test results

Water-absorption Test Results

Results in Fig. 7 indicate that the increment of slag in concrete decreased water-absorption capacity. The use of 22.5% slag showed lower water absorption compared to control concrete. The reasons may be the filler and more CSH gel made using waste slag in the concrete (Gururaj et al., 2015). Slag provided more CSH gel in compact concrete compared to the control, as has been specified in XRD. Further increment in slag increased the percentage of water absorption. This was due to more fines in concrete, reducing the bond and compaction (Dhanesh et al., 2019).

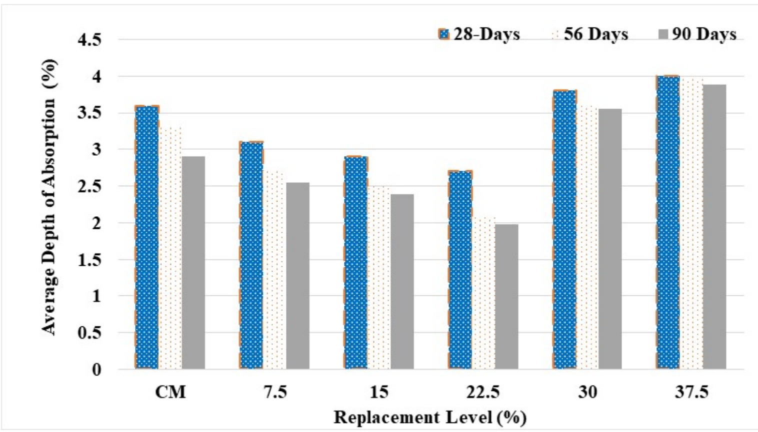


Figure (7): Water-absorption test results

Water-permeability Test Results

Results shown in Fig.8 indicate that the permeability of the slag sample decreased with increased % of waste. The minimum penetration depth was obtained at 22.5% replacement of cement with slag. The results indicated that slag enhanced the behavior of concrete due to its volatile and filler effect (Nakum et al., 2015). More increment of slag increased the depth of penetration, because reducing the content of cement in concrete and reducing the CSH gel reduced bonding (Dinakar et al., 2013).

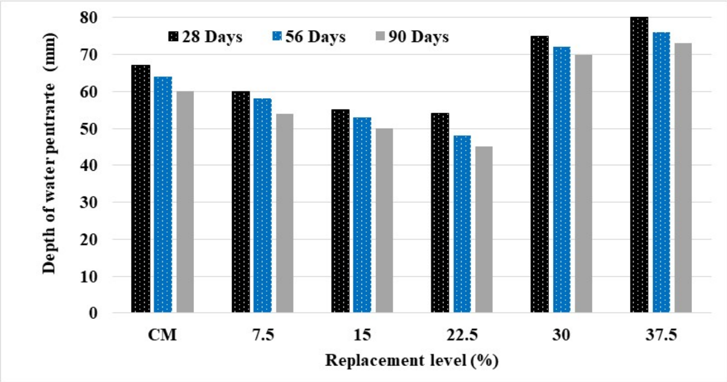


Figure (8): Water-permeability test results

RCPT Results

From Fig. 9, the values of charge in Coulombs (C) can be observed. From the results, it is shown that charges reduced with increased value of slag in concrete. The value of minimum charges was obtained at 22.5% slag incorporated in concrete. Charges help in deciding the permeability of concrete with different percentages of slag. The reason is that fine particles and strong bonding between particles reduced the charges (Ipieca et al., 2015). More increments in slag increased the value of the charges and increased the permeability of the mixes. Increased fines in concrete in terms of slag which replaced cement reduced bonding (Mashaly et al., 2018).

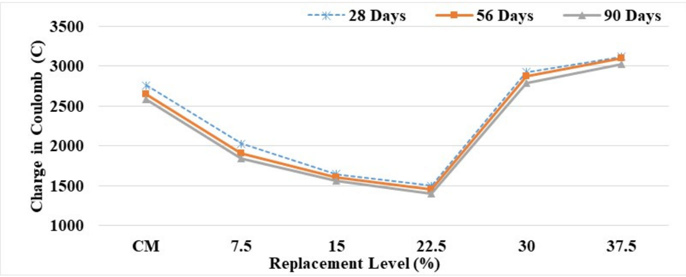


Figure (9): Test results of RCPT

Carbonation Test Results

Fig. 10 indicates the values of depth from the CO_2 attack. The results show that less penetration was found by increasing the value of iron slag in concrete. The minimum penetration was obtained at 22.5% slag incorporated in concrete. Penetration helps in deciding the quality of concrete with different percentages of slag. Fine particles and strong bonding between particles reduced the charges (Pade and Guimaraes, 2007). More increment of slag in concrete increased the charges and increased the penetration. Increased fines in concrete in terms of slag which replaced cement reduced bonding (John and John, 2013).

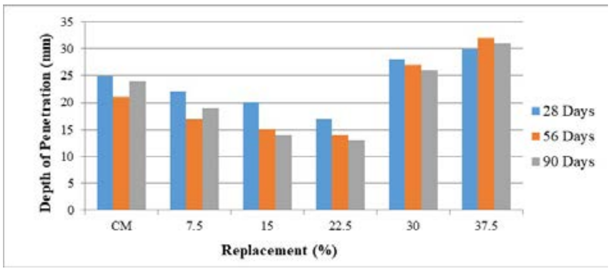
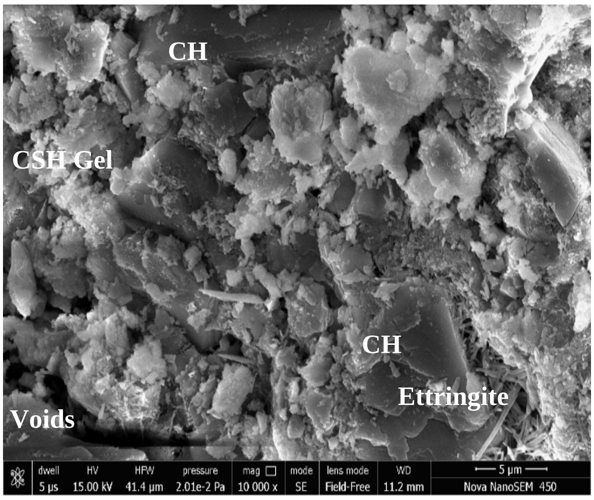


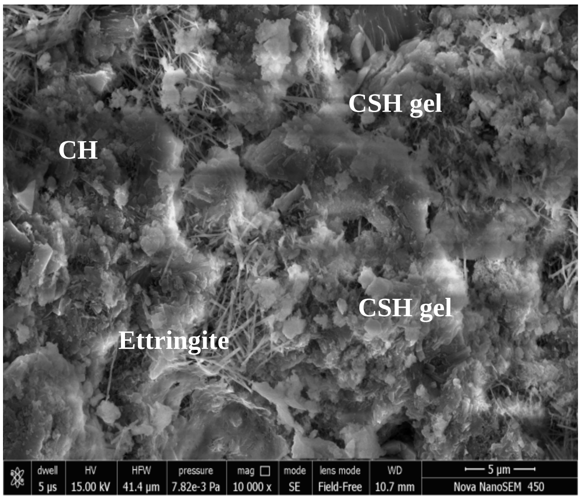
Figure (10): Carbonization-test results

Micro-structural Results

SEM Analysis

Fig.11 shows the SEM analysis of the control and iron slag specimens. In this analysis, cement and aggregate phases were observed. Images showed that incorporation of 22.5% slag represented a more compact and denser mortar phase than the control concrete. Results indicated an improved behavior of slag concrete due to utilization of fines as cement. One more reason was the specific gravity of iron slag which is higher in comparison to the raw material (El-Didamony and Amer, 2016).



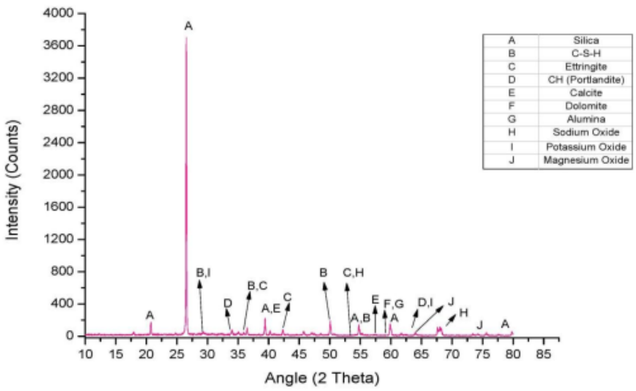


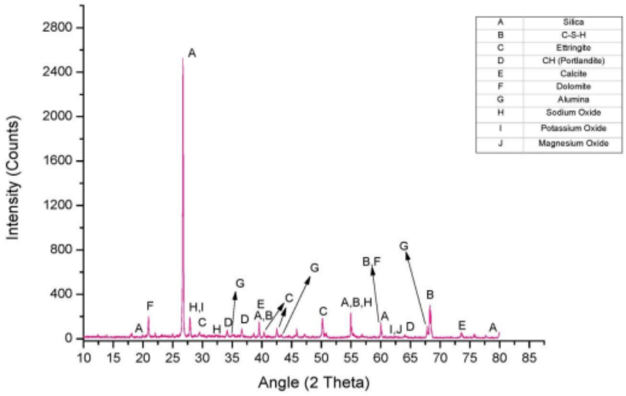
Control Concrete **(b) 22.5% Iron Slag**

Figure (11): Test results of SEM analysis

XRD Analysis

Fig.12 shows the peaks of control concrete and slag concrete at 22.5%. It is perceived that the peak of CH in slag concrete was minor compared to the peak of CH in the control concrete mix. The reason was the transformation of CH into CSH gel, which was formed due to additional silica present in the slag powder (Alizadeh et al., 2003). Further increment reduces the formation of CSH gel in slag-made concrete due to less amount of calcium oxide available for the reaction.





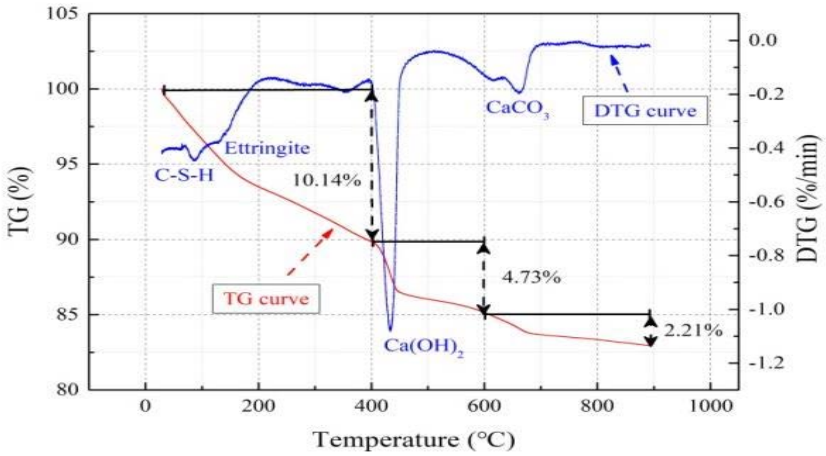
(a) Control Concrete **(b) Slag at 22.5%**

Figure (12): Test results of XRD analysis

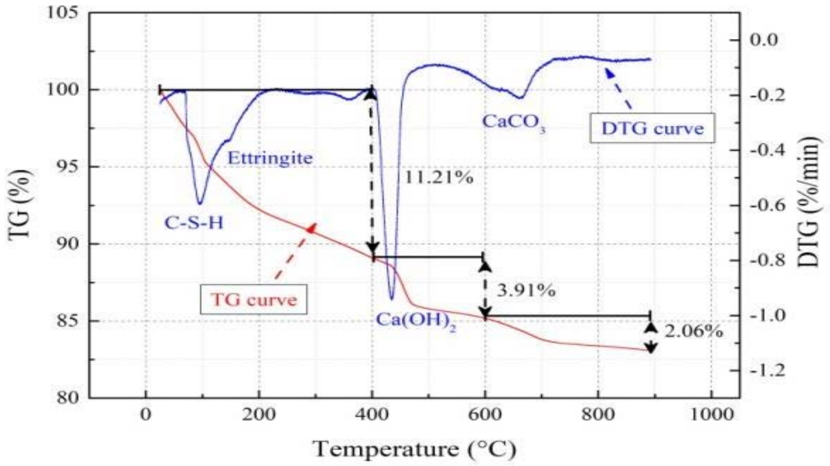
TGA Analysis

The analysis results of TGA–DTG conducted on the control and blended mixes of concrete at different curing ages are shown in Fig. 13. The mass defeat between room temperature and 100°C, as shown in the TGA curve, was due to the retreat of free water from the concrete (Nadeem & Pofale, 2012). The mass loss that occurred between 100°C and 150°C was attributed to the loss of combined water due to the dehydration of the C–S–H gel, which in fact corresponded to the first strong endothermic peak in the DTG curve. Between 150°C and 200°C, the mass loss of ettringite occurred.

Meanwhile, between 400°C and 450°C, the mass loss of CH occurred during dehydration and a second strong endothermic peak was observed in the DTG curve (Harison et al., 2014). Between 600°C and 650°C, the third endothermic peak appeared in the DTG curve. At this time, $CaCo_3$ began to decompose and be decarbonized into CaO and CO_2 overflows.



(a) Control Concrete



(b) Slag at 22.5%

Figure (13): Test results of TGA analysis

CONCLUSIONS

Iron slag has a great potential to turn out to be a partial substitute for cement in concrete production. The following conclusions may be drawn based on the outcomes of the test results:

- The slump value of slag-added concrete was found less compared to the control mix. At higher replacement levels, the slump value achieved was almost near to 50 mm. Workability for all mixes was maintained, so that they can be used in light reinforced sections.
- Up to the replacement of a certain amount of cement with slag, the compressive and flexural strengths were improved. The maximum compressive strength was achieved at 22.5% replacement, which is 23% higher than that of the control concrete. The trend for 28-d, 56-d and 90-d strength was similar for compressive and flexural strengths.
- Water absorption and penetration of the cement-replaced specimens were lower than in the control sample. The minimum penetration and water absorption were observed at 22.5% replacement. Higher replacement increased the permeability and water absorption of respective concrete.
- IS 15658-2006 has specified the permissible limit of

6% for water absorption of all types of concrete mixes.

- Test results from RCPT indicated that the concrete mix with 22.5% slag has a low value of permeability (1438 C). This is due to the slower reactivity of glass and granite, generating pozzolanic C-S-H. This additional C-S-H gel contributes to the compact micro-structure, improving the overall strength properties of concrete.
- Less penetration of CO_2 indicates the better quality of concrete. Higher incorporation of slag increased the penetration of CO_2 in concrete.
- SEM and XRD analyses show that the density and packing of concrete matrix are proper in 22.5%-slag sample, which enhances its results as compared to control concrete.
- TGA results were in support of slag-made concrete. They indicated that 22.5% replacement of cement by slag improved the features of concrete.

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