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Research Article

Sustainable Approach for Linz-Donawitz Slag Waste as a Replacement of Cement in Concrete: Mechanical, Microstructural, and Durability Properties

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Linz-Donawitz (LD) slag, solid waste produced during steel manufacture in basic oxygen furnace, is difficult when it comes to handling and disposal and has very limited utilization. To increase its reusability, the suitability of supplanting cement with LD slag in concrete was examined. To study the impact of partially replacing cement with LD slag on strength, microstructure, and durability of concrete, more than 150 samples were cast. The test results reveal that the highest compressive and flexural strengths were attained at 20% LD slag replacement and, beyond that, the strength decreased. The hydration products detected by X-ray Diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) were calcium silicate hydrate (C-S-H), ettringite, and calcium aluminosilicate hydrate (C-A-S-H). The scanning electron microscope (SEM) images of binary cement concrete showed denser microstructure and lesser voids. The sulphuric acid resistance, electrical resistivity, and carbonation resistance tests done reveal that acceptable durability could be achieved when cement is replaced by LD slag. It is deduced that LD slag can be utilized in partially replacing cement to achieve the desired strength. This research gives another comprehension of simultaneously managing steel industry waste for sustainable development and contributing added advantages to the economy.

1. Introduction

The substantial growth in the Indian steel industry in recent times has led to massive and varied solid wastes generation. Blast furnace slag and steel furnace slag (LD slag and electric arc furnace slag) comprise the majority of waste slag generated during the manufacturing of iron and steel, respectively. Based on the ore feed, LD slag is about 15–20% of the steel produced and blast furnace slag accounts for about 30–55%, respectively [1]. The discharged slag requires plenty of land for dumping and is hazardous to the surroundings as it has certain pollutants leaching to soil. India produces about 12 million tons of steel slag per annum but the rate of utilization is lower than 20 [2]. Unlike steel furnace slag, blast furnace slag has been successively used in construction industry. In the current practice, the utilization of steel slag is primarily done as filling material in construction of roads [3, 4] or as partial replacement of coarse aggregates [5–7]. Improved compressive strengths and acceptable durability were reported. Ahmedzade and Sengoz [8] reported that better cohesive strength, high stiffness, and electrical resistance were observed when coarse aggregate was partially replaced by steel slag. Cement is one of the most utilized building materials in construction across the world, while the cement manufacturing process emits high CO_2 to the environment. Use of LD slag as a cementitious component instead of aggregate in concrete would boost its reuse and will reduce the cost of construction and production of greenhouse gases significantly. The main constraint with the usability of LD slag is the high amount of free MgO and free CaO content, which leads to volumetric changes [9–11]. At the same time, owing to calcined materials present in LD slag, its potential to be used as a partial substitution of OPC (ordinary Portland cement) may turn out beneficial for the environment and economy [12]. Researchers proposed LD slag as a pozzolanic material to be used for manufacturing concrete due to the existence of a definite quantity of cementitious minerals such as C_2S, C_3S, C_4AF, and C_2F found in it [13–16]. Research done on Portland cement clinker production made by the addition of slag in the raw feed indicated acceptable mineralogical characteristics for lower replacement ratios [17, 18]. Some studies have shown that LD slag cement slows the early hydration of blended mixes [19, 20] but could achieve acceptable physical properties [21, 22] at lower replacement ratio. The addition of LD slag decreased the workability of concrete [23]. Specifically, the literature available for the microstructural properties is extremely diminutive. Very few researches have indicated that concrete with acceptable durability properties can be achieved when LD slag is utilized as material for cement replacement [24]. As per the understanding of authors, research focusing on the effect of utilization of LD slag as cementitious component on the properties of concrete is limited. Thereafter, efforts have been made to conclude the findings concerning the addition of various proportions of LD slag as cement substitute on the hardened state of concrete. A parametric experimental study has been conducted to investigate the effect on the mechanical, microstructural, and durability properties of the concrete containing LD slag. The study of hydration products and the microstructure of the binary mix will lead to the comprehension of development of strength and durability of concrete. A good understanding will thus lead to its increase as a construction material, thereby imparting benefits to the environment and economy.

The prominent objective of the present work is to study the outcome of partially replacing cement with LD slag on the mechanical, microstructural, and durability properties of concrete. Different proportions of slag were added to this study for comparison purposes. An attempt to investigate the pozzolanic behavior of LD slag when mixed with cement on the hardened properties is done. The mechanical properties of the binary cement concrete were evaluated through compressive strength tests and flexural strength tests. The effects of addition of LD slag on the hydration products and microstructure of concrete were also investigated through SEM, XRD test, and FTIR test. The nonevaporable water content of the concrete samples was found to evaluate the degree of hydration. Several tests such as sulphuric acid attack, carbonation depth, ultrasonic pulse velocity, and electrical resistivity were done to evaluate the durability property of concrete. Laboratory test results from the concrete specimens were then compared and final conclusions were drawn.

2. Experimental Programme

2.1. Materials. In the present study, Grade 43 OPC as per IS: 8112 (2013) [25] was used. The chemical composition of LD slag procured from Bhilai Steel Plant, Bhilai, India, was determined using XRF (X-ray fluorescence) analysis as listed in Table 1. The collected samples were crushed in a pulverizer and then sieved with 90 μm sieve size. The images of OPC and LD slag powder are depicted in Figures 1(a) and 1(b). The specific gravity of LD slag and OPC used was 3.25 and 3.10, respectively. The specific surface area of cement and LD slag was found to be 3435 cm^2/g and 4734 cm^2/g, respectively. The particle size distribution curve of LD slag and cement is as shown in Figure 2. From the figure, it can be seen that the LD slag particles are finer than OPC as indicated by the specific surface area. Regional river sand belonging to zone II was utilized as fine aggregate. Coarse aggregate of a maximum size of 20 mm was used in accordance with IS 383 (1970) [26]. The specific gravity of coarse aggregate and fine aggregate was found to be 2.64 and 2.56, respectively. The method suggested by Mason [27] was used for calculating the alkalinity of slag, which was found to be 2.60. Therefore, it can be considered as a material for partial cement replacement as its alkalinity is higher than 1.80 [15].

2.2. Mixture Proportions and Methods. To deduce the effect of replacing OPC partially with LD slag (up to 30%), a total of 4 mix proportions were considered in the present work for comparative study. Various tests were done to examine the impact of LD slag on mechanical, microstructural, and durability properties of concrete when utilized as a partial cement substitute.

Cube moulds of 100 mm size were used. Size of the concrete beam specimen taken was 100 mm ∗ 100 mm ∗ 500 mm as the aggregate size is less than 19 mm. A water/binder ratio of 0.40 was fixed and a set of 12 samples were cast for each mix. The specimens were prepared by initially compacting the mixture manually and then by the use of the vibrating table. Moulds were demoulded after 24 hours and the cubes were then cured for periods of 7, 28, 56, and 90 days, respectively, in water at room temperature. Concrete mixes were made in accordance to IS 10262 (2009) [28], which have been given in Table 2, where C is cement and S is LD slag with the numbers following them being the percentage of each. The compressive strength tests were performed on compression testing machine (CTM) of 2000 kN capacity and the flexural strength tests were performed on flexural testing machine (FTM) of 100 kN capacity. The beams were subjected to four-point loading conditions and the load was applied at the rate of 180 kg/min. The flexural test checks the ability of nonreinforced beam to withstand the failure in bending. After every 7, 28, 56, and 90 days, the compressive and flexural strengths of samples were tested and the average value of three samples in MPa was recorded at each curing period.

The microstructure of the sample was analyzed by scanning electron method (SEM). Gold coating was done over the samples to obtain SEM images using the ZEISS scanning electron microscope machine. XRD for paste specimens was performed in a PANalytical X'Pert platform in order to analyze hydration characteristics. For preparing the cement pastes, 400 g of mixtures (LD slag and cement) with 100 ml of water was used to examine the hydration products. Curing for 28 days was done at a temperature of $27 \pm 2^{\circ}C$. Analysis was systematically done between 500 and 4000 cm^{-1}. The test was performed by Bruker FTIR spectrometer at room temperature of $27 \pm 2^{\circ}C$. The samples of concrete cured for 28 days were used for FTIR analysis.

Table 1: Chemical composition of ordinary Portland cement and LD slag.

Components	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	$Na_2O + K_2O$	P_2O_5	MnO
OPC	67.51	16.70	4.65	3.76	1.66	—	0.37	2.35	—
LD SLAG	45.98	15.88	1.40	17.04	6.67	—	—	1.8	1.32

(a)

(b)

Figure 1: Images of materials used. (a) Ordinary Portland cement. (b) LD slag.

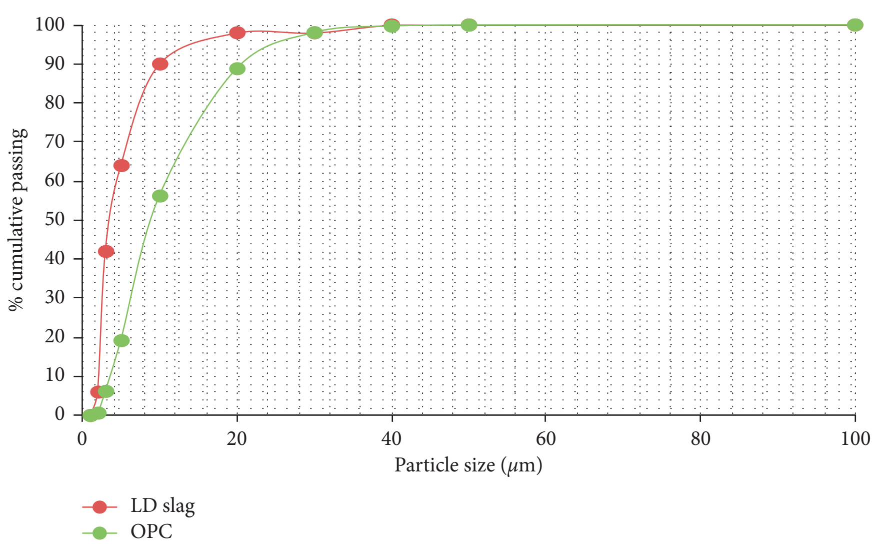

Figure 2: Particle size distribution of OPC and LD slag.

Table 2: Concrete mix proportions in kg/m^3.

Mix designation	Cement (OPC)	Steel slag	Fine aggregate	Coarse aggregate	Water	Water/binder ratio
CM	383	0	667.2	1223.2	153.2	0.4
C90S10	344.7	38.3	667.2	1223.2	153.2	0.4
C80S20	306.4	76.6	667.2	1223.2	153.2	0.4
C70S30	268.1	114.9	667.2	1223.2	153.2	0.4

To evaluate the degree of hydration, the samples hydrated for 7 and 28 days, respectively, were tested for their nonevaporable water content (Wn). The samples (hardened pastes) were heated at a temperature of 65°C for a period of 24 hours in an oven. They were then heated in a muffle furnace at 1000°C for 2.5 hours. Due to the temperature difference of 65°C and 1000°C, the loss of mass per gram of binder is defined as the Wn content [29].

The soundness of LD slag was determined using Le-Chatelier apparatus in accordance with IS 5514 (1996) [30] to check the volumetric stability of LD slag owing to the existence of free calcium and magnesium oxides.

To analyze concrete qualitatively, a nondestructive UPV test was performed on the concrete cubes. Evaluation of internal cracks, the uniformity, and cohesiveness of the mix was done by using the IS 13311 (1992) [31] guidelines. The concrete quality is considered excellent if ultrasonic pulse velocity values are above 4.5 km/sec. The test was performed on the concrete samples of all varying mixes cured for 7, 14, and 28 days, respectively.

The acid attack test was performed by preparing sulphuric acid solution, 5% by volume in acid-resistant tanks. After 28 days of curing, the initial mass of cubes was noted before immersing in the acid solution. The pH of the solution was kept constant throughout immersion period. After 4 weeks, the difference in samples weight was measured by first cleaning them with tap water and then drying.

For the accelerated carbonation test, cubes were subjected to accelerated carbonation curing conditions at 30°C, 70% relative humidity, and 5% CO_2. After a curing period of 28 days at room temperature, the samples were kept inside the carbonation chamber and the parameters mentioned above were maintained throughout the test. Phenolphthalein was used as an indicator. It was applied on the fractured concrete surface to depict the carbonated zone. After exposure of 4 weeks to carbonation curing, the carbonation depth of samples was determined.

The electrical resistivity test was done using Giatec RCON™ concrete electrical resistivity meter. The test cubes cured for 7, 14, and 28 days were placed in between two plates placed parallel to each other. On each side, a wet sponge is placed on top of the cube and current is passed through the electrodes. The average value of three cubes was taken to determine the bulk electrical resistivity of the concrete.

3. Results and Discussion

3.1. Strength. To assess the mechanical behavior of the manufactured concrete, compressive and flexural strength tests were performed on cubes and beams, respectively, with varying curing time. Figures 3 and 4 show the results for the compressive and flexural strength of the different types of concrete made. It can be noted from Figure 3 that the mixture of LD slag and OPC improves the strength of concrete. Initially slag behaves as an inert material and is not chemically active as compared to OPC, also known as physical filling effect. The compressive strength of samples C90S10 after 7 days was found to be 90.7%, which was near to that of the control sample after curing for 28 days. Meanwhile, when compared to the control sample, the compressive strength of samples C80S20 and C70S30 increased from 86% to 74.4% and from 92.5% to 85.9%, respectively, after curing for 28 days. However, after 90 days of curing, the compressive strength values of samples C90S10 and C80S20 exceeded that of control concrete. Similar trend was noticed for the flexural strength results. It also depicts

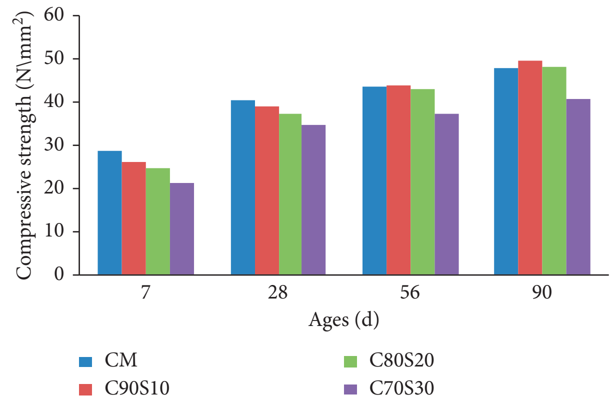

FIGURE 3: Compressive strength of concrete mixes at various curing ages.

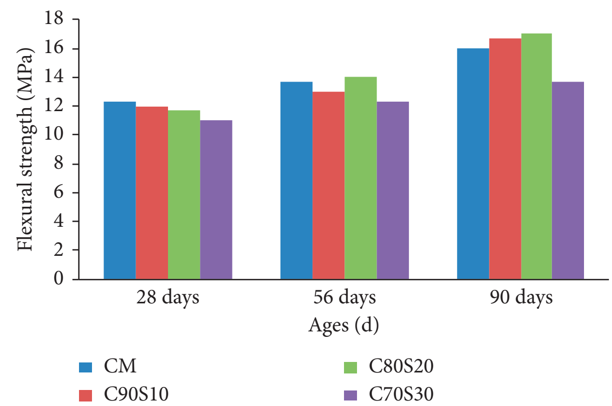

FIGURE 4: Flexural strength of concrete mixes at various curing ages.

that increasing the percentage of LD slag above 20% decreased the compressive and flexural strength by 15.27% and 14.56%, respectively, even after 90 days of curing. From sustainability point of view, sample C80S20 is preferred over sample C90S10 as higher replacement ratio of slag is used without compromising the strength parameter. From the results, it can be said that LD slag has high reactivity at later ages and the strength is thus improved due to the formation of secondary C-S-H gel.

3.2. Hydration Products and Microstructure of the LD Slag Pastes

3.2.1. SEM and XRD Analysis. The XRD result of hardened cement paste with 20% LD slag after being cured for 28 days is depicted in Figure 5. From the test, the primary products of hydration observed were C-S-H ($Ca_4(SiO_3)_3(OH)_2$), portlandite ($Ca(OH)_2$), and ettringite ($Ca_6Al_2(SO_4)_3{\cdot}32H_2O$). The RO (CaO–FeO–MnO–MgO solid solution) and C_2F phases are considered as inert mineral components that have nil or lower degrees of reaction. The main mineralogical phases that remain unhydrated are identified as C_2S and C_3S even after 28 days of curing. The morphologies of hardened samples of control and binary mixes were also determined.

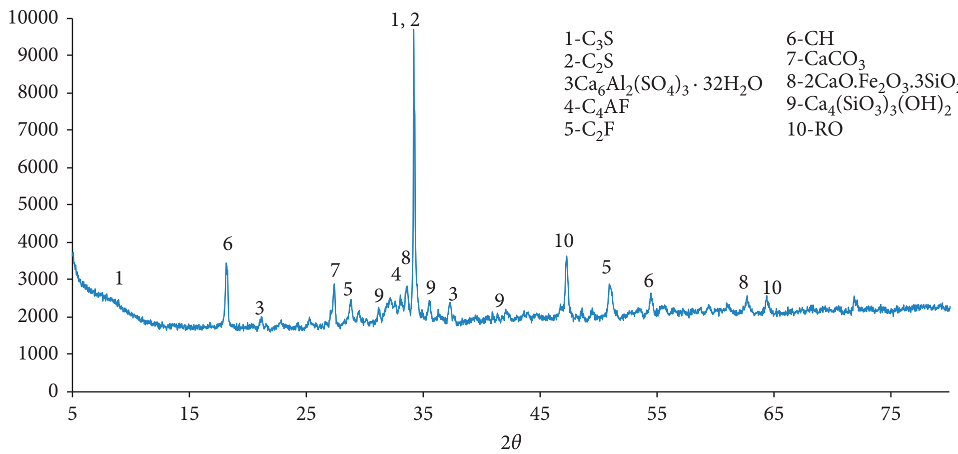

Figure 5: XRD of cement with 20% steel slag, hydrated at 28 days.

Concrete samples cured for 7 and 28 days were tested by SEM to study the microstructure. As depicted in Figures 6(a) and 6(b), after standard curing for 28 days, as compared to OPC, the voids in binary mix C80S20 are lesser, indicating reduced pore volume. Also the structure is denser, which in turn reduces the permeability of concrete making it more durable. Because of this denser structure and thicker ettringite needles, higher compressive strength is achieved at later ages of curing.

3.2.2. FTIR Analysis. Figure 7 gives the FTIR spectra of LD slag concrete sample C80S20 after 28 days of curing. IR spectra can identify components to S-O vibrations of SO_4^{2-} with absorption band around 602.21 cm^{-1} as gypsum ($CaSO_4{\cdot}2H_2O$) [32]. In the concrete samples, the absorption bands at 1478.52 cm^{-1} and 3395.26 cm^{-1} match with calcium carbonate and the O-H bond stretching associated with calcium hydroxide formation, respectively. The Al-O-H bending at around 700.26 cm^{-1} is attributed to the formation of ettringite (AFt) [33]. The vibrations in the 1082.35 cm^{-1} region are assigned to the presence of Si-O stretching, which confirms the generation of calcium silicate hydrate (C-S-H) [34]. This observation is in good agreement with the XRD analysis.

3.3. Nonevaporable Water Content. Figure 8 depicts the nonevaporable water (Wn) measurements of the hardened samples. It can be noticed that the Wn content of both cement pastes with and without LD slag increased with curing time. As compared to LD slag, more hydration products are produced by cement. Therefore more replacements of cement lead to a lower content of Wn. The Wn content of binary cement paste samples C90S10, C80S20, and C70S30 was only 6.76%, 5.86%, and 4.95% at 7 days, whereas that of control paste was 7.76% at 7 days. This clearly indicates that the pozzolanic activity of binary mix is lower at early ages. After 28 days, out of all the binary mixes, the Wn content of sample C80S20 was found to increase the most by 48.14%, whereas an increase of 41.25% was observed for control concrete. A greater growth rate than that of control cement paste was observed, indicating that as the curing period increases the reactivity of slag increases at a faster rate than that of cement.

3.4. Soundness. The volumetric instability of LD slag is subjected to the existence of free (unhydrated) lime (CaO) or free MgO. The soundness of the cement paste samples prepared with LD slag was tested using Le-Chatelier process as per IS 4031 (part 3) 1988 [35]. The soundness of the binary paste prepared by replacing cement with LD slag up to 30% was observed to be 3 mm. For 43 grade OPC cement, the maximum expansion allowed according to IS 8112 (2013) [25] is 10 mm. Therefore, the value obtained even at the highest replacement ratio tested is well below the specified limits.

3.5. Ultrasonic Pulse Velocity. The concrete pulse velocity after being cured for 28 days is as shown in Figure 9. The UPV test was done to find the internal compactness of concrete in terms of homogeneity, porosity, and other defects. It was found that the UPV values of all the binary mixes were lower than the control concrete after 7 days of curing but increased as period of curing increased. This is possible due to the slower hydration kinetics or reactivity of LD slag. A linear increase in the UPV values was observed for all the mixes as the curing period increases. However, it was observed that, for the mix C70S30 even at 28 days, the UPV values were slightly lower than the control mix but even then they were higher than 4.5 km/sec. Improved particle packing achieved due to finer LD slag particles can be attributed for such result. Therefore it can be considered as excellent concrete as per IS 13311 (part 1) 1992 [31]. It is an indication of formation of denser microstructure and reduced porosity.

3.6. Sulphuric Acid Attack. From the results, as depicted in Figure 10, it can be noticed that all concrete samples experience mass loss when subjected to 4 weeks of immersion in 5% sulphuric acid solution. The possible reason for mass

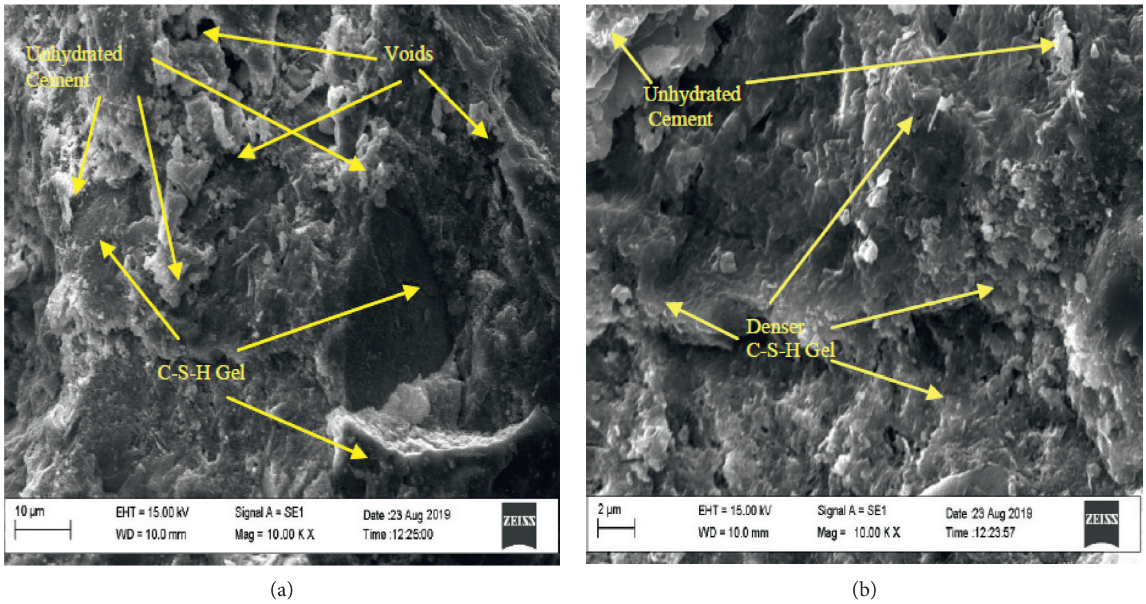

FIGURE 6: SEM micrographs. (a) Control sample CM. (b) Binary sample C80S20.

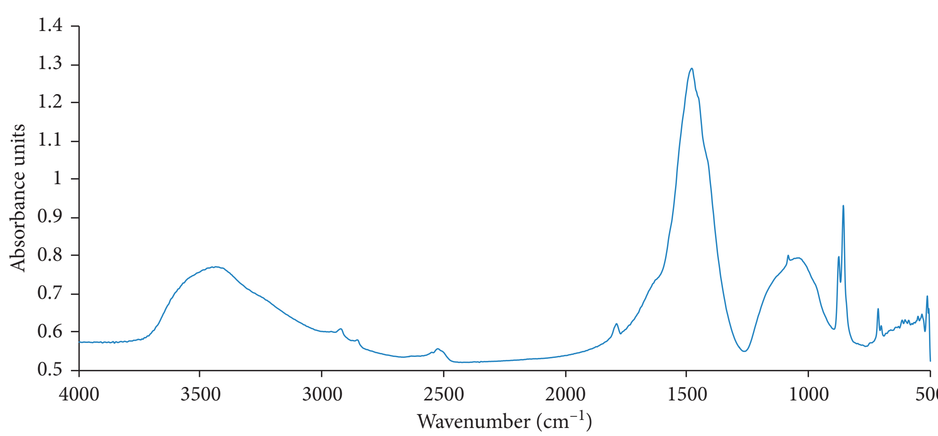

FIGURE 7: FTIR spectra for concrete sample C80S20 after 28 days of curing.

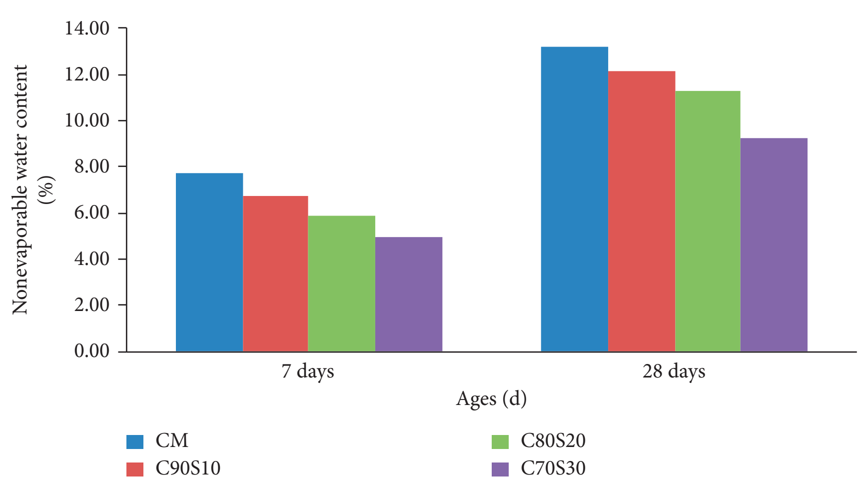

FIGURE 8: Nonevaporable water content of cement pastes.

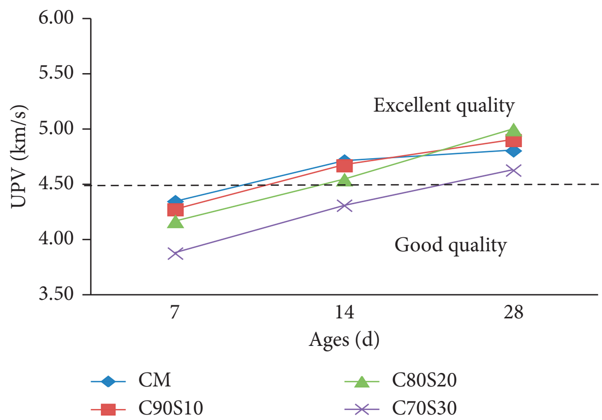

Figure 9: Ultrasonic pulse velocity of concrete mixes.

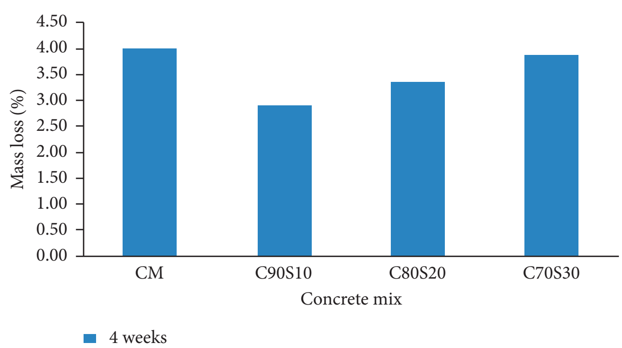

Figure 10: Mass changes of concrete specimens immersed in 5% sulphuric acid solution for 4 weeks.

loss and consequent formation of porous structure is the lowering of pH. In the acidic environment, $Ca(OH)_2$ gets dissolved and an unstable C-S-H structure undergoes decalcification. The effect of H_2SO_4 attack decreases as the replacement of LD slag increases. The mixes with binary addition are more resistant to sulphuric acid attack by 27.5%, 16.25%, and 3% for samples C90S10, C80S20, and C70S30, respectively, as compared to the control specimen. Due to binary mix, lower permeability of concrete is achieved as a result of more refined pore structure. The durability of concrete is thus increased as the resistance to acid attack gets improved due to the addition of LD slag.

3.7. Carbonation Depth. Figure 11 displays the result of carbonation depth of average of three specimens of each mix kept under accelerated curing conditions for 28 days. It was observed that increasing the replacement ratio of LD slag has minute effect on the carbonation resistance of concrete as compared to control specimen. After standard curing of 28 days, when compared to control specimen, the depth of carbonation of mix C90S10 shows no significance deviation, whereas the mix C80S20 shows comparable results. It is in agreement with results found by other researchers [24]. The possible reason for this is that although the lime content of LD slag is lower than that of OPC, it is sufficiently high for the pH to be maintained. As an outcome of carbonation, so

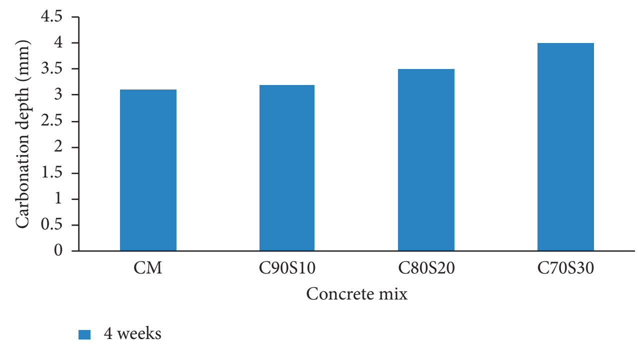

Figure 11: Carbonation depth of concrete mixes for an exposure period of 4 weeks.

Table 3: Relationship between concrete resistivity and corrosion rate (ACI 222R-01 2001) [36].

Resistivity (kΩ-cm)	Corrosion rate
>20	Low
10 to 20	Low to moderate
5 to 10	High
<5	Very high

formed $CaCO_3$ densifies the microstructure by filling up the pores. Thereafter, the permeability of concrete reduces and thus hinders the further penetration of CO_2 into the concrete. Hence, on the basis of observed results, it can be said that partially replacing cement up to 20% of LD slag has no negative effect on the concrete and acceptable durability can be achieved.

3.8. Electrical Resistivity. The electrical resistance of concrete is indicative of the properties of its microstructure. The electrical resistance to the movement of ions within the microstructure of concrete indirectly indicates the presence of voids and connectivity of pores. The durability of concrete can therefore be reflected through the values of electrical resistivity. Uniaxial method of testing was adopted and the electrical resistance is calculated as follows:

$$\rho = \frac{RA}{L}, \tag{1}$$

where ρ is electrical resistivity; R is electrical resistance; A is cross-sectional area perpendicular to current; and L is electrical path length of sample.

The relationship between the corrosion rate and concrete resistivity is available in ACI Committee 222 report (ACI-222-R-01 2001) [36] as depicted in Table 3. According to Figure 12, it can be said that, at 7 days, as the replacement ratio of LD slag increases, the electrical resistivity of concrete decreases. However, after 28 days of curing, denser structure is observed even at higher replacement levels for all the specimens. The values of electrical resistivity for all the samples were found to be in the category of low rate of corrosion as per ACI Committee 222, indicating no detrimental effect on the durability of concrete when replacement of cement by LD slag is done.

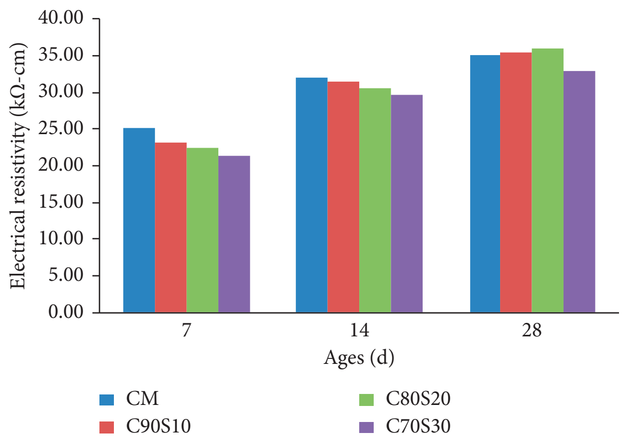

Figure 12: Electrical resistivity of concrete mixes.

4. Conclusions

The present experimental study reveals the effects of partially replacing cement by LD slag on strength and durability of concrete. The conclusions were drawn from the study on the basis of experimental results and analyses are as follows:

(1) Increasing the content of LD slag above 20% results in decreasing the compressive and flexural strength of the concrete. The compressive strength of sample C80S20 was found to be comparable to control sample after curing for 28 days. However, higher reactivity of slag is observed as the curing period increases and thus the compressive strength exceeds the strength of control concrete at 90 days. Latent hydraulic property of slag can be attributed for such result.

(2) Hydration products of hardened binary sample confirm the presence of Aft, C-S-H gel, and C-A-S-H. This forms a denser microstructure with lesser voids as compared with the control concrete as depicted by SEM morphologies.

(3) The soundness of samples reduced due to the incorporation of LD slag. Nevertheless, the values of soundness of LD slag-cement paste samples were all within specified limits as per IS 8112 (2013).

(4) Concrete samples made with LD slag display better resistance to sulphuric acid. This may be attributed to reduced porosity and better particle packing of pores.

(5) The depth of carbonation after 28 days of curing was not significantly influenced by the addition of LD slag. Acceptable durability is achieved as diffusion of CO_2 decreases due to improved permeability of concrete at later ages.

(6) The bulk electrical resistivity value falls under the head of low rate of corrosion for all the samples, indicating denser microstructure and lower permeability of concrete made with LD slag. The durability of concrete is thus improved.

Considering compliance with codal provisions of all important properties, LD slag as a partial replacement of cement can be used. It results in a very cost-effective and sustainable development with no compromise in strength parameters. Further analysis of the steel slag also may be conducted based on its metallic Fe content.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

There are no conflicts of interest.

Acknowledgments

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