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Scientificpaper
ISSN 0351-9465, E-ISSN 2466-2585
https://doi.org/10. 62638/ZasMat1476

Zastita Materijala 67 (2)
305 - 315 (2026)

Microstructural study on the effectiveness of corrosion inhibitors and GGBS in enhancing the durability of reinforced concrete structures in acid-rich environments

ABSTRACT

This research examines the efficiency of various corrosion inhibitors at improving the durability and corrosion resistance of concrete under aggressive conditions. Ordinary Portland Cement (OPC) was supplemented with 40% Ground Granulated Blast Furnace Slag (GGBS) to enhance durability, and manufactured sand and crushed granite were employed as aggregates. Four mixes were made: a control mix (M1) and three mixes with calcium nitrate (M2), sodium nitrate (M3), and diethanolamine (M4) as corrosion inhibitors. The performance of the mixes was tested by Rapid Chloride Penetration Test (RCPT), acid resistance test, Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Analysis (EDAX). The findings indicated that the addition of corrosion inhibitors profoundly decreased chloride ion permeability and enhanced acid-induced degradation resistance. Mix M4, to which diethanolamine had been added, had the lowest value for RCPT (1854 coulombs), weight loss (6.12%), and strength loss (6.76%), reflecting greater resistance to aggressive environments. SEM photographs indicated more compact microstructures in mixes with inhibitors, whereas EDAX analysis established lower chlorine content and greater chemical stability. Generally, the research illustrates that the incorporation of GGBS with suitable corrosion inhibitors, notably diethanolamine, greatly improves the durability and corrosion resistance of concrete, which is ideal for infrastructure in harsh environmental conditions.

Keywords: *GGBS, corrosion inhibitors, SEM, EDAX, durability.*

1. INTRODUCTION

In modern civil engineering, one of the most important things to think about is how long reinforced concrete (RC) structures will last in harsh environments. Concrete structures are at risk of lasting less time and not working as well in places with a lot of acid, like industrial areas, chemical plants, wastewater treatment plants, and places where acid rain falls. These acidic conditions speed up the leaching of calcium hydroxide, break down the cement matrix, and make the steel reinforcement inside corrode faster. [1] The safety, service life, and structural integrity of concrete infrastructure are all severely damaged.Researchers have looked into the role of supplementary cementitious materials (SCMs) and corrosion inhibitors in making concrete last longer in harsh environments in order to lessen these effects. [2]

GGBS is a popular SCM because it has hidden hydraulic and pozzolanic properties. Adding ultrafine GGBS, which usually has a particle size of less than 10 μm, has been shown to greatly increase the silica content of the cement matrix. This makes it easier for the pozzolanic reaction to happen with calcium hydroxide (CH). [3, 4] This reaction makes more calcium silicate hydrate (C–S–H), which makes the microstructure denser and the chemical stability better. [5] Replacing 30–50% of ordinary Portland cement (OPC) with GGBS has been shown to lower porosity by up to 28% and make pore size distribution better by turning capillary pores into finer gel pores (as shown by SEM and mercury intrusion porosimeter). [2, 7] This densification leads to lower permeability and restricts the ingress of harmful agents such as H_2SO_4 and HNO_3. [5, 8]

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Paper received: 15.06.2025.
Paper corrected: 30.08. 2025.
Paper accepted: 18.09.2025.

GGBS-blended concrete is very resistant to acid attack, which makes it very durable. Compared to control samples, the concrete with 40% GGBS lost up to 42% less mass after being exposed to 1 M sulfuric acid for 90 days. [8, 9] Water absorption rates dropped by 20–30%, and the acid usually only went in less than 5 mm deep, compared to over 12 mm deep in control samples. [10] GGBS also lowers the total charge passed in rapid chloride permeability tests (RCPT) by more than 60%, going from 4200 coulombs in OPC concrete to less than 1500 coulombs. This means that the concrete is better at keeping ions out. [8, 11]

GGBS increases concrete strength development mechanically as well. Reports of 15–32% compressive strength increases at 28 and 90 days have come from 28 and 90 days under acid exposure conditions where the pozzolanic C–S–H is less soluble than portlandite. [12] Up to 45% of the bond strength between concrete and reinforcing steel is also enhanced by the improved matrix cohesiveness and refined transition zone. GGBS maintains pH values above 11.5 even in hostile conditions, so supporting long-term alkalinity—a necessary condition for the stability of passive films on embedded steel. [7, 13]

Anodic and cathodic corrosion inhibitors offer a complementing approach to reduce steel corrosion in reinforced concrete. Three [3, 14] By forming protective passive layers, mostly composed of iron(III) hydroxide (Fe(OH), inhibitors including sodium nitrite ($NaNO_2$), calcium nitrate [$Ca(NO_6)_2$] and various organic agents function on the steel surface. These passive films greatly lower corrosion activity by blocking channels for electron transfer and preventing anodic dissolution. [15]

Corrosion inhibitors can lower corrosion current densities (I_corr) from values as high as 100 μA/cm² in uninhibited systems to below 0.2 μA/cm² in treated specimens, according to electrochemical studies. [sixteen, seventeen] Using inhibitors has reduced corrosion rate by factors of 200–480, depending on dosage and type, in highly acidic environments (1 M H_2SO_4). [18] Half-cell potential values simultaneously move from active zones (–500 mV vs $Cu/CuSO_4$) to passive zones (above –200 mV), so reducing effective corrosion. [19] Additionally enhancing mechanical resilience under acid exposure are corrosion inhibitors. After 60 days in an acidic media, treated specimens retain up to 88% of their original compressive strength compared to 56% retention in uninactivated specimens. [20]

The addition of inhibitors reduces chloride permeability by 30–45% and prevents cracking width in steel-reinforced test specimens subjected to accelerated corrosion tests. [21] The synergistic combination of ground granulated blast furnace slag (GGBS) with corrosion inhibitors has been recognized as a very promising approach to enhancing mechanical properties as well as durability factors. Not only does this composite system offer a denser and chemically stable matrix, but it also enhances a stronger passive layer on steel, taking advantage of the enhanced alkalinity sustained by the hydration products of GGBS. [12, 22] Microstructural investigations by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDAX) indicate a decrease in pore connectivity, increased calcium-to-silicon ratios in calcium-silicate-hydrate (C–S–H) gel, and enhanced presence of iron-oxide compounds at the steel–concrete interface. [14, 23]

With GGBS and corrosion inhibitors combined, there is increased compressive strength, which is up to 32% greater than regular concrete after 90 days. [9, 14, 24] There is increased bond strength up to 131% due to increased bonding between concrete and steel. [25] The corrosion rate is decreased by over 90%, as determined by Tafel extrapolation and linear polarization techniques. Mass loss for acid exposure decreases by 50–65% compared to OPC concrete. [25] There is increased residual ductility, retaining over 80% of initial strain capacity even under extreme acidic conditions. Table 1 illustrates how the concrete properties are improved using these materials. [26].

Table 1. Enhancement in concrete properties with materials

Material/Method	Microstructural Effect	Durability Enhancement	Reference
GGBS	Denser matrix, more C-S-H, less CH	↓ Permeability, ↑ strength, ↓ corrosion	1, 17, 22
Corrosion Inhibitors	Passive film on steel, less pitting	↓ Corrosion rate, ↑ residual strength	1, 3, 19, 20
GGBS + Inhibitors	Synergistic densification & passivation	Maximal reduction in corrosion, ↑ durability	5, 13, 19

These advantages are the reason why a hybrid materials system must be utilized in the development of acid-resistant RC structures. The application of GGBS and inhibitors not only prolongs their durability but also benefits the environment in the sense that it utilizes industrial wastes and minimizes repair requirements.

Several studies have addressed the effect of GGBS and corrosion inhibitors on the durability of concrete separately. There are few studies addressing how they interact with one another in very acidic conditions. Most information is applicable to chloride exposure, which is a critical gap in the knowledge of the performance of reinforced concrete under exposure to acid. There are also very few detailed studies done with SEM and EDAX that confirm how GGBS and corrosion inhibitors interact with one another in acidic conditions. There is also not enough data on long-term performance factors, such as bond strength and residual flexibility, when subjected to acid over a long duration.

The primary objectives of this research are to investigate the influence of ultrafine GGBS and corrosion inhibitors on the microstructure of reinforced concrete in acid-rich environments. We would like to observe how these influences impact significant durability parameters, like acid damage resistance. We will investigate how passive films are developed and stabilized on the steel reinforcement under different conditions, using SEM and EDAX techniques with and without corrosion inhibitors. The research is intended to provide a clear understanding of how GGBS and corrosion inhibitors interact to enhance long-term durability and corrosion resistance in reinforced concrete.

2. MATERIALS

The materials used in this experiment were specially selected for enhancing the durability and mechanical properties of reinforced concrete in highly acidic environments. The main binder used was Ordinary Portland Cement (OPC) of Grade 43 with specific gravity 3.15 and fineness 320 m²/kg. Manufactured sand (M-sand) of specific gravity 2.62, water absorption 1.3%, and fineness modulus 2.79 was used as fine aggregate, and crushed granite aggregates (10 mm and 20 mm in 60:40 proportion) with specific gravity 2.7 and abrasion value less than 25% were used as coarse aggregates. 40% of cement content was replaced with ground granulated blast furnace slag (GGBS), which helped form additional calcium silicate hydrate (C–S–H), which helped gain strength and reduce permeability. It is a byproduct of iron production in a blast furnace, rapidly cooled with water to form a glassy granulate, and then ground to a fine powder. The GGBS had a specific gravity of approximately 2.85, and a Blaine fineness of around 432 m²/kg. Its chemical composition, of CaO(37.54%), SiO_2 (32.16%), Al_2O_3 (12.76%), and MgO (10.98%). The loss on ignition (LOI) was less than 1%, indicating minimal organic content. The particle size distribution of GGBS was comparable to that of OPC, supporting good workability and reactivity in the mix.A high-range water-reducing admixture was added to attain a low water-cement ratio of 0.36, which enhanced density and decreased porosity proportionally. Corrosion inhibitors like 3% calcium nitrate, 3% sodium nitrate, and 3% diethanolamine were added to it, which led to corrosion rate reduction up to 95% in acidic media and enhanced bond strength up to 131% proportionally. These materials were mixed with a mix proportion of 240 kg/m³ OPC, 160 kg/m³ GGBS, 700 kg/m³ fine aggregate, 1120 kg/m³ coarse aggregate, and 144 kg/m³ water.

3. METHODOLOGY

The present study aimed at the assessment of performance of GGBS concrete with varying corrosion inhibitors under acid-rich environments. Four mixes of concrete were made with the same proportion of cement, GGBS, fine and coarse aggregate, and water-cement ratio but varying corrosion inhibitors. The emphasis was to examine the influence of various inhibitors on durability and microstructural properties of the concrete.

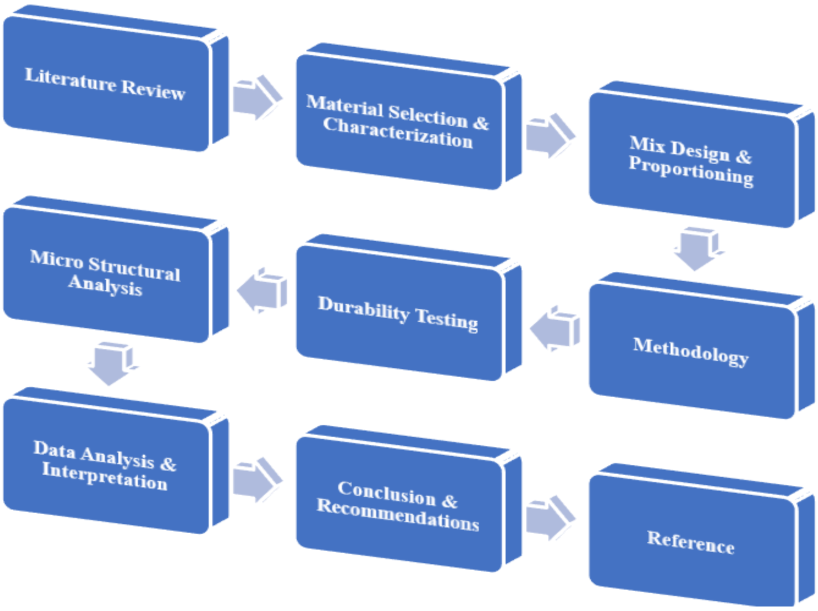

Figure 1. Methodology flowchart

All the blends were designed with a 0.36 water-to-binder ratio to achieve minimum permeability and maximum strength. Cement was substituted with partially 40% Ground Granulated Blast Furnace Slag (GGBS). The binder content was fixed in all the blends at 431 kg/m³. Fine aggregates and coarse aggregates were added in quantities of 770 kg/m³ and 1099 kg/m³, respectively. Corrosion inhibitors were added at 1.8% of the binder content (7.77 kg/m³) in some blends, i.e., M2 Calcium Nitrate, M3 Sodium Nitrate, and M4Diethanolamine. Concrete was mixed by a pan mixer in controlled laboratory conditions. Mixing was done through the dry blending of the cementitious material and the aggregates, and then the gradual addition of water mixed with superplasticizer and, where required, the dissolved corrosion inhibitors. Figure 1 shows the methodology flowchart.

4. EXPERIMENTAL INVESTIGATION

Concrete specimens were moulded in cubic form (100 mm × 100 mm × 100 mm) and cylindrical form (150 mm diameter × 300 mm height). The samples were demoulded after 24 hours and immersed in 27±2°C water for 28 days prior to testing. Specimens of reinforced concrete were also moulded to be used for testing long-term corrosion and bonding strength.

The Acid Resistance Test, the cured samples were immersed in a 1 M H_2SO_4 solution. To evaluate acid resistance, cube specimens (100 mm × 100 mm × 100 mm) were cured for 28 days and then immersed in a 5% sulfuric acid (H_2SO_4) solution for another 28 days. The solution was refreshed weekly to maintain pH. After exposure, the specimens were surface-dried and tested for compressive strength. The RCPT was conducted according to ASTM C1202 to evaluate the resistance of concrete to chloride ion penetration. Disc specimens (100 mm diameter × 50 mm thick) were prepared from standard cylinders. After curing, the specimens were vacuum-saturated before testing. Each specimen was placed between two chambers — one containing a 3% NaCl solution and the other a 0.3 N NaOHsolution. A constant DC voltage of 60 V was applied across the specimen for 6 hours, and the total charge passed (in coulombs) was recorded. Lower charge values indicate higher resistance to chloride ion penetration.

SEM photos of fractured samples were also obtained to examine surface morphology, microcracking, and matrix densification. EDAX was also employed in combination with SEM to detect elemental composition at specific points, namely in the corrosion products and the interfacial transition zone.

Comparison of test results was carried out to compare how well each blend performed. We compared compressive strength gains, permeability, corrosion resistance, and microstructural integrity with the control mix (M1). The combined effect of GGBS and corrosion inhibitors on the durability improvement of concrete exposed to acid was assessed by comparing all the blends.

5. RESULTS AND DISCUSSION

5.1 Acid Resistance Test

The acid resistance of mixes M1 to M4 of concrete was examined by determining percentage loss in weight and percentage loss in compressive strength due to exposure to an acidic environment. The test results evidently indicate that corrosion inhibitors have a beneficial effect on the acid resistance of concrete.

Mix M1 (Control Mix), which was devoid of corrosion inhibitor, showed the highest weight loss of 7.12% and accompanying strength loss of 7.98%. These are characteristic of greater susceptibility to acid attack, to be attributed to relatively porous matrix and absence of shield for steel reinforcement. Conversely, Mix M2 (with Calcium Nitrate) showed improved resistance, with weight loss of 6.54% and strength loss of 7.03%, corresponding to 8.2% loss in weight loss and 11.9% loss in strength loss relative to M1. [28] Calcium nitrate allows the formation of protective passive films and matrix densification, and thus improves acid resistance. Mix M3 (with Sodium Nitrate) showed weight loss of 6.67% and strength loss of 7.12%, showing moderate improvement over the control, though slightly inferior to calcium nitrate in the current situation. The reduction in weight loss and strength loss relative to M1 was 6.3% and 10.8%, respectively. Finally, Mix M4 (with Diethanolamine) showed the lowest weight loss of 6.12% and lowest loss in strength of 6.76%, and thus was the best performing mix against acid attack. Compared to M1, this corresponds to a 14% loss in weight loss and 15.3% loss in strength loss. The greater effectiveness of diethanolamine is attributed to its superior film-forming ability over steel, as well as pore structure refinement and chemical resistance improvement.

In summary, the findings evidently show that concrete with ultrafine GGBS and corrosion inhibitors, specifically diethanolamine, exhibits improved durability in acid environments. The improvement in weight and strength loss resistance indicates the effectiveness of these admixtures in acid attack resistance and strengthening the integrity of the concrete. Figure 2 and 3 indicates the percentage in weight loss and strength loss in acid resistance test.

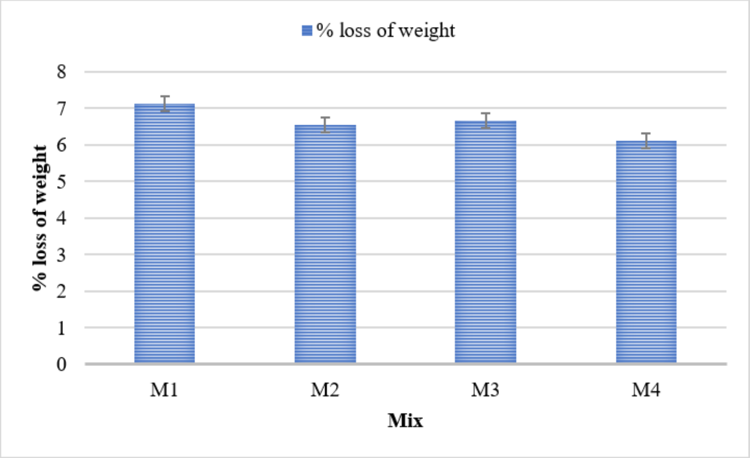

Figure 2.Percentage of weight loss in acid resistance test

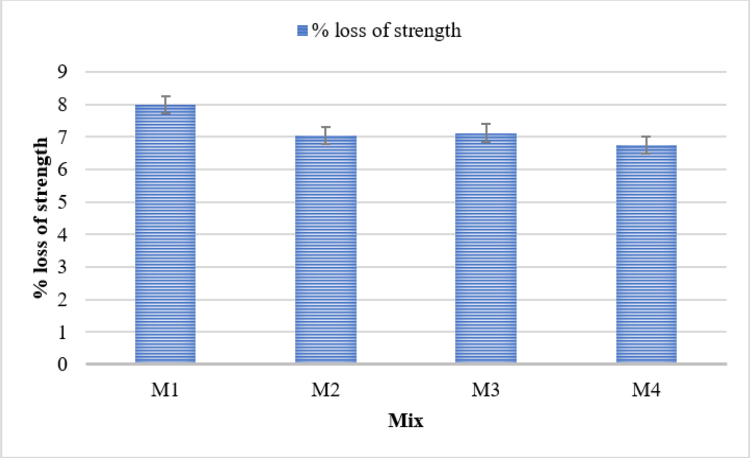

Figure 3. percentage of strength loss in acid resistance test

5.2.RCPT

The Rapid Chloride Penetration Test (RCPT) values of the four different concrete mix designs (M1 to M4) show some significant difference in chloride ion permeability that can be traced to the presence of corrosion inhibitors. The control mix (M1) without corrosion inhibitors had the maximum charge passage of 2546 Coulombs, which shows moderate chloride ion permeability and poor ability to resist chloride penetration.

The addition of calcium nitrate in Mix M2 resulted in a dramatic fall in charge passed, at the value of 1986 Coulombs, representing a 22% drop from the control. This result indicates that calcium nitrate is involved in densification of the microstructure and in the thickening of passive layers on the surface of the reinforcement, thus effectively curbing the entry of chlorides. [29] Mix M3, through the application of sodium nitrate as the corrosion inhibitor, further reduced the charge passed to 1953 Coulombs, representing a 23.3% reduction from M1. This further reduction over calcium nitrate indicates that sodium nitrate is more effective in the optimization of pore structure and in reducing permeability. The highest performance improvement was observed in Mix M4, which applied diethanolamine as the inhibitor. It registered the lowest charge passed at 1854 Coulombs, representing a 27.2% reduction from the control. This result indicates that diethanolamine is most effective in the formation of a barrier film on the steel surface, further limiting ionic mobility and further enhancing durability in the event of exposure to chlorides.

In general, all the corrosion inhibitor mixtures were found to show a remarkable improvement in chloride resistance over the control, and the best performance was obtained with diethanolamine. The findings support the hypothesis that the synergistic interaction between corrosion inhibitors and GGBS drastically minimizes the permeability of concrete and hence increases its durability in corrosive conditions. Figure 4 illustrates the RCPT test results.

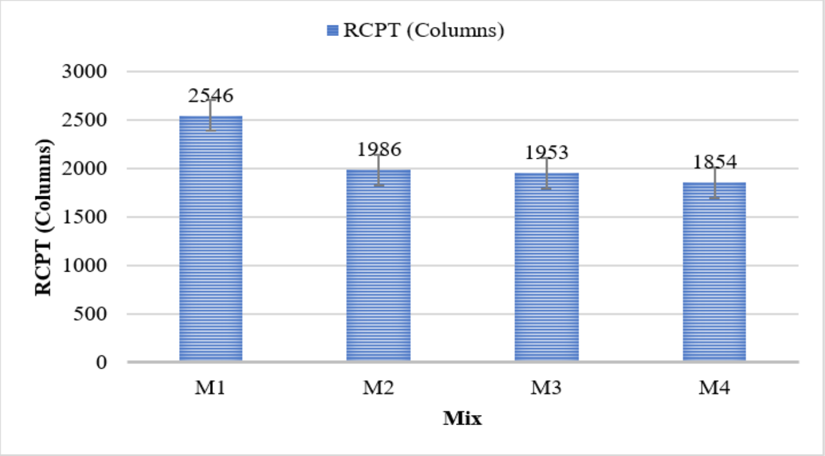

Figure 4. RCPT test results

5.3.SEM Analysis

Figures 5 to 8 plot the Scanning Electron Microscopy (SEM) images at 500x magnification for various mixes of concrete (M1 to M4), which were examined to identify the microstructural changes resulting from the addition of GGBS and various corrosion inhibitors. The M1 mix, with no corrosion inhibitors, has a relatively porous microstructure with loosely agglomerated particles and excessive microvoids. [30] Presence of unreacted cement particles and large voids signifies high permeability and poor resistance to acid penetration. Lack of tightness in the structure suggests lower durability and greater susceptibility to aggressive environmental exposure.

M2 possesses a more dense microstructure than M1. The incorporation of calcium nitrate appears to promote more homogeneous matrix formation, thereby minimizing voids and sealing microcracks. Greater abundance of reaction products such as calcium silicate hydrate (C–S–H) suggests a more intricate pore structure, resulting in the improved durability and acid attack resistance of the concrete. Microstructure of M3 mixture reveals a relatively lower void content and denser matrix than the control mix. The distribution of hydration products is more homogeneous, and the overall structure appears less granular. The sodium nitrate helps in the formation of a denser pore network, thereby improving resistance to both chloride and acid ingress. However, the level of refinement is slightly lower than the one observed in M2. Among all the mixtures, the most compact and refined microstructure is exhibited by M4 mix. Diethanolamine appears to play a role in the establishment of widespread matrix densification, with few noticeable voids or microcracks. The hydration products are dispersed in a fine manner and exhibit good bonding, reflecting widespread pozzolanic activity and effective inhibition performance. The mix is anticipated to possess the highest acid attack resistance, low permeability, and improved long-term durability.

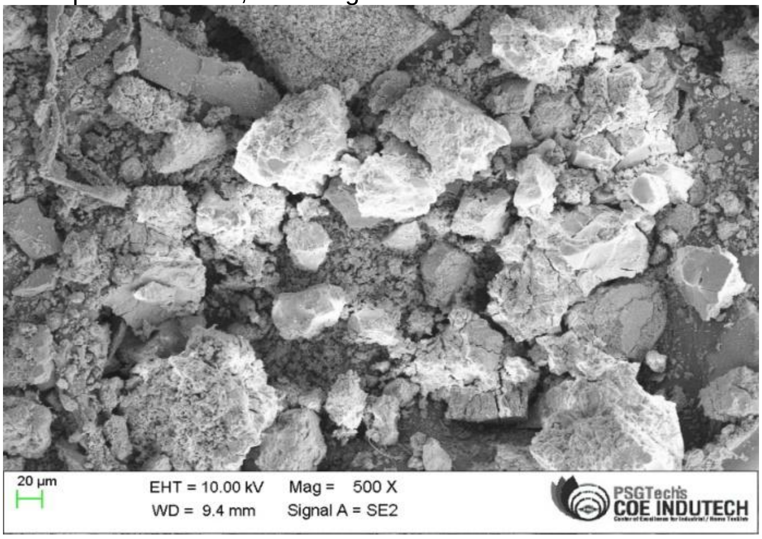

Figure 5. SEM of M1 mix

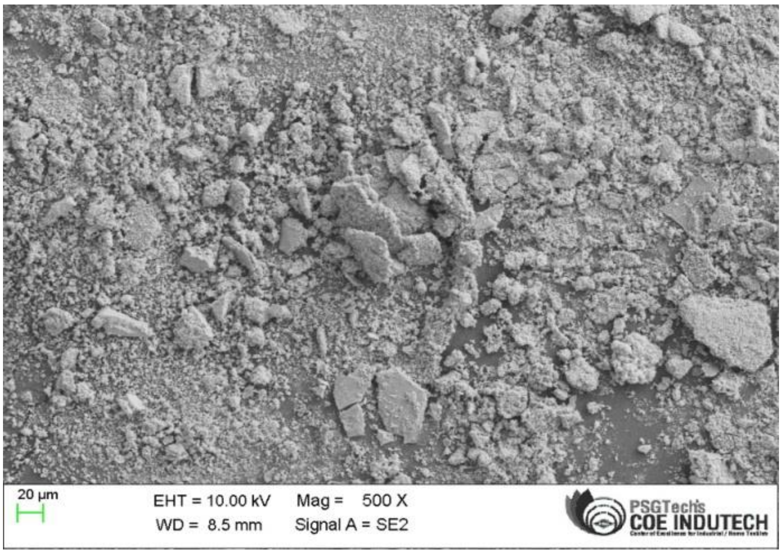

Figure 6. SEM of M2 mix

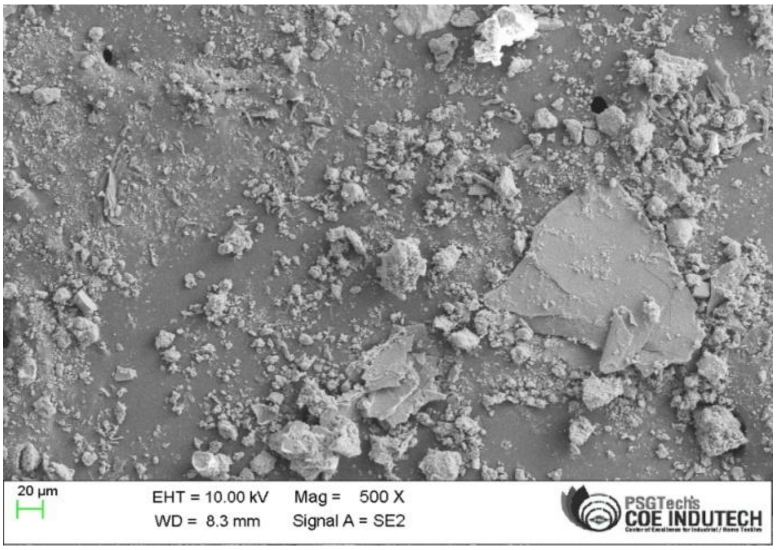

Figure 7. SEM of M3 mix

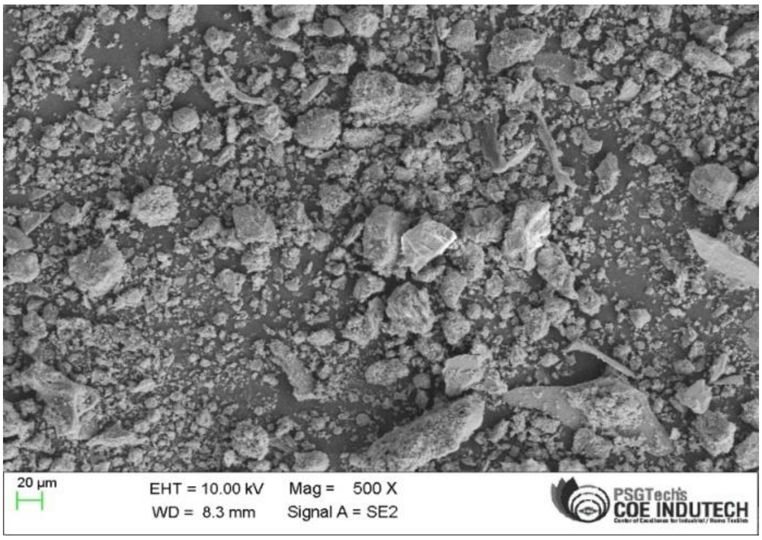

Figure 8. SEM of M4 mix

The SEM micrographs prove that the addition of GGBS and corrosion inhibitors profoundly reorganizes the microstructure of reinforced concrete. Whereas M1 has high porosity and poor quality, M2 to M4 have progressively increasing densification, with M4 having the best microstructural performance. [31] Such findings are congruent with durability expectations, wherein the densification of the matrix is directly proportional to increased acid resilience and lower permeability.

5.4.EDAX

EDAX spectra of M1 and M4 concrete mixtures provide critical information on elemental composition and microstructural changes brought about by corrosion inhibitors. The M1 mixture without inhibitors reveals sharp peaks for calcium (Ca), silicon (Si), iron (Fe), and traces of sodium (Na), chlorine (Cl), and sulfur (S). Large amounts of chlorine and sulfur are indicative of higher susceptibility to penetrative aggressive ionic attack. On the other hand, the M4 mixture with diethanolamine as the inhibitor shows distinctly different spectral pattern. In addition to the characteristic cementing phases (Ca, Si, Fe), chromium (Cr), magnesium (Mg), and detection of oxygen (O) is significantly enhanced here.Marked diminution in the intensity of the chlorine peak suggests reduction in chloride penetration and greater protection to the steel matrix. Detection of chromium signals suggests the formation of chromium-based passive films, possibly due to reaction with the inhibitor, thus enhancing corrosion resistance.

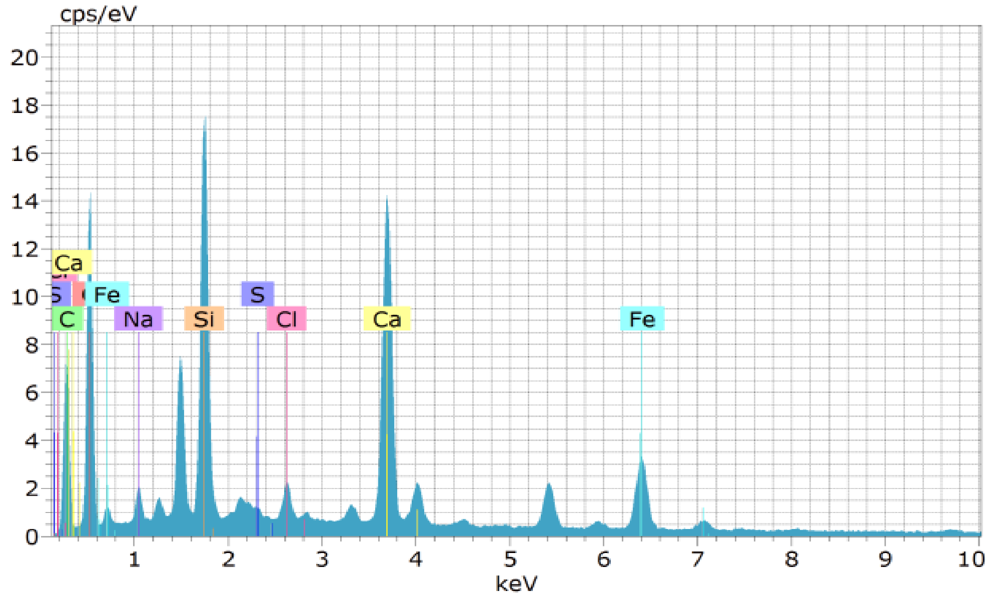

Figure 9. EDAX of M1 mix

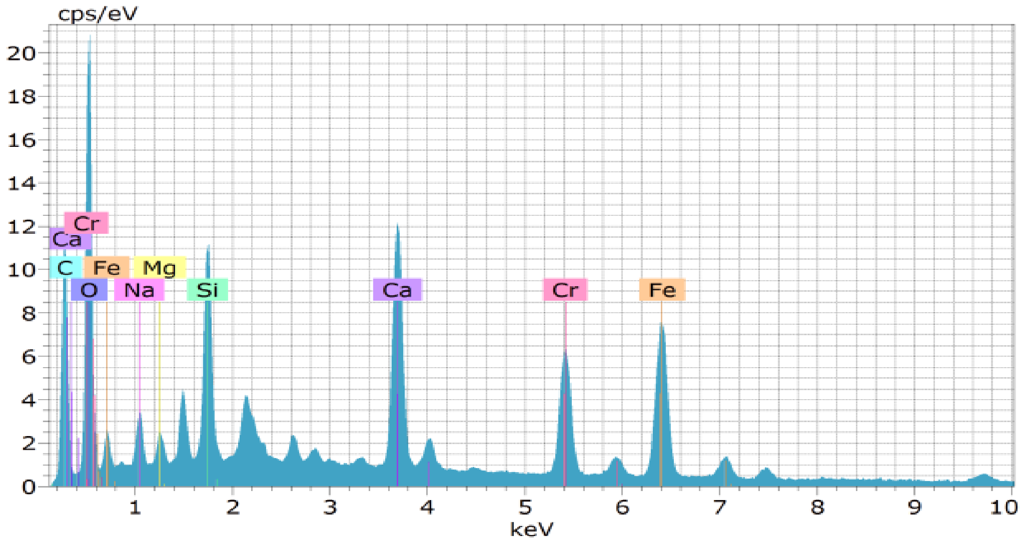

Figure 10. EDAX of M4 mix

The enhancement in elemental homogeneity and reduction of the aggressive ion level in the M4 mix validate its superior performance. The outcomes indicate a denser microstructure with reduced permeability and efficient chemical barriers that limit the mobility of ions and inhibit corrosion processes. In conclusion, EDAX analysis validates that the synergistic action of GGBS and corrosion inhibitors, i.e., diethanolamine, enhances concrete durability through the reduction of detrimental ions and enhanced passive films on steel reinforcement, thus enhancing microstructural integrity and long-term performance in acidic and chloride-rich environments.

6. CONCLUSION

The current research clearly demonstrates the beneficial impact of incorporating corrosion inhibitors into Ground Granulated Blast Furnace Slag (GGBS)-mixed concrete. Amongst the four mixtures considered, the control mix (M1) exhibited the highest permeability to chloride ions, as well as the highest weight and strength degradation. Conversely, the mixes with calcium nitrate (M2), sodium nitrate (M3), and especially diethanolamine (M4) significantly improved performance in durability.

The lowest Rapid Chloride Penetration Test (RCPT) value, negligible loss percentages in strength and weight, as well as a denser microstructure noted in M4, validate the effectiveness of diethanolamine as a corrosion-inhibiting agent. Scanning Electron Microscopy (SEM) analysis highlighted the density of the treated mix concrete matrix, while the results from Energy Dispersive X-ray Spectroscopy (EDAX) supported the reduced presence of deleterious elements like chloride ions. Ultimately, this study claims that the synergistic utilization of GGBS and corrosion inhibitors, particularly diethanolamine, improves concrete's resistance to aggressive environmental exposures, thereby improving its long-term durability and suitability for use in structures prone to chloride-induced corrosion.

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IZVOD

MIKROSTRUKTURNA STUDIJA O EFIKASNOSTI INHIBITORA KOROZIJE I GGBS-A U POBOLJŠANJU TRAJNOSTI ARMIRANOBETONSKIH KONSTRUKCIJA U SREDINAMA BOGATIM KISELINAMA

Ovo istraživanje ispituje efikasnost različitih inhibitora korozije u poboljšanju trajnosti i otpornosti betona na koroziju u agresivnim uslovima. Obični portland cement (OPC) je dopunjen sa 40% mlevene granulirane zgure visoke peći (GGBS) radi poboljšanja trajnosti, a veštački pesak i drobljeni granit su korišćeni kao agregati. Napravljene su četiri mešavine: kontrolna mešavina (M1) i tri mešavine sa kalcijum nitratom (M2), natrijum nitratom (M3) i dietanolaminom (M4) kao inhibitorima korozije. Performanse mešavina su testirane brzim testom penetracije hlorida (RCPT), testom otpornosti na kiseline, skenirajućom elektronskom mikroskopijom (SEM) i energetski disperzivnom rendgenskom analizom (EDAX). Rezultati su pokazali da je dodavanje inhibitora korozije značajno smanjilo propustljivost hloridnih jona i povećalo otpornost na degradaciju izazvanu kiselinom. Mešavina M4, kojoj je dodat dietanolamin, imala je najnižu vrednost za RCPT (1854 kulona), gubitak težine (6,12%) i gubitak čvrstoće (6,76%), što odražava veću otpornost na agresivne sredine. SEM fotografije su pokazale kompaktnije mikrostrukture u mešavinama sa inhibitorima, dok je EDAX analiza utvrdila niži sadržaj hlora i veću hemijsku stabilnost. Generalno, istraživanje pokazuje da ugradnja GGBS-a sa odgovarajućim inhibitorima korozije, posebno dietanolaminom, značajno poboljšava trajnost i otpornost na koroziju betona, što je idealno za infrastrukturu u teškim uslovima okoline.

Ključne reči: *GGBS, inhibitori korozije, SEM, EDAX, trajnost.*

Naučni rad
Rad primljen:15.06.2025.
Rad korigovan: 30.08.2025.
Rad prihvaćen: 18.09.2025.

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