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https://doi.org/10.5109/7323349

出版情報：Proceedings of International Exchange and Innovation Conference on Engineering & Sciences (IEICES). 10, pp.779-785, 2024-10-17. International Exchange and Innovation Conference on Engineering & Sciences
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Producing Magnesium Hydroxide Coating from Seawater to Prevent Hydrogen Sulfide-induced Corrosion in Concrete

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Abstract: *The anaerobic nature of concrete structures in sewage, industrial, and agricultural sectors result in the proliferation of sulfate-reducing bacteria. These bacteria release hydrogen sulfide through sulfate reduction processes, rendering the exposed concrete structure to hydrogen sulfide-induced corrosion. While commercial magnesium hydroxide coatings ($Mg(OH)_2$) are being used as a conventional method, this study explored using seawater-derived $Mg(OH)_2$ as an alternative sustainable protective coating. Three set-ups were tested: uncoated concrete, seawater-derived $Mg(OH)_2$ coated concrete, and commercially available $Mg(OH)_2$ coated concrete. Each setup had four replicates and was exposed to 4M sulfuric acid for four days. To evaluate the coating's effectiveness, the compressive strength of the specimens was assessed. The tests revealed that seawater-derived $Mg(OH)_2$ coated specimens did not outperform those coated with commercially available $Mg(OH)_2$. However, they demonstrated superior performance compared to the uncoated specimens. Overall, this highlighted the potential of seawater-derived $Mg(OH)_2$ as a sustainable alternative for mitigating hydrogen sulfide-induced corrosion.*

Keywords: Seawater; Sustainable Protective Coating; Magnesium Hydroxide; Wastewater; Concrete

1. INTRODUCTION

Hydrogen sulfide-induced corrosion in concrete commonly occurs in environments exposed to anaerobic conditions, including sewer, industrial, and agricultural settings [1]. Their anaerobic nature allows sulfate-reducing bacteria (SRB) to thrive and convert sulfate (SO_4^{2-}) present in the environment into hydrogen sulfide through the process of sulfate reduction. This typically occurs in sections where the flow of water is stagnant or slow-flowing [2]. In the presence of moisture, hydrogen sulfide is converted to sulfur dioxide (SO_2) and sulfur trioxide (SO_3) through a metabolic process called chemosynthesis. This is conducted by the *Thiobacillus thiooxidans* bacteria to be used as their source of energy. These sulfur compounds then dissolve in water to produce sulfuric acid, which causes corrosion to the exposed concrete [3]. However, current mitigation methods such as polyurethane, epoxy tar coal pitch, and commercially available magnesium hydroxide pose detrimental impacts on the environment [4].

Given that the construction sector has been one of the leading contributors to greenhouse gas (GHG) emissions, accounting for 37% in the year 2022, sustainable alternatives have continuously developed to mitigate the detrimental impacts of construction on the environment [5-7]. In this context, seawater emerges as a promising sustainable alternative for extracting magnesium hydroxide compared to its commercially available counterpart due to its abundance and high concentration of magnesium ions. Seawater constitutes about 97% of the Earth's water sources and contains approximately 1,300 parts per million (ppm) or 1.29 g/L of magnesium (Mg^{2+}) ions, making it a promising source of magnesium hydroxide [8].

As such, this study extracted magnesium hydroxide from seawater for use in the production of protective coatings against hydrogen sulfide-induced corrosion in concrete structures. Through this study, sustainable alternatives for mitigating hydrogen sulfide-induced corrosion may be established.

2. MATERIALS

2.1 Material Properties

For this study, 100 mm x 100 mm x 100 mm concrete specimens were cast. Table 1 presents the composition of the concrete design mix. All materials used for the concrete mix comply with DPWH standards for non-reinforced concrete sewers, storm drains, and culvert pipes [9]. This standard was chosen to accurately replicate the conditions of such concrete structures that are exposed to hydrogen sulfide-induced corrosion. A target compressive strength and slump of 28 MPa and 80 mm, respectively, were chosen for this study.

Table 1. Composition of concrete mixture for $0.01m^3$ of concrete

Material	Type	Composition
Cement	Portland Cement Type 1T (Pozzolan + Limestone)	4.52 kg
Water	Tap Water	2.26 kg
Coarse Aggregates	Crushed Gravel	9.95 kg
Fine Aggregates	Concrete Sand (washed)	6.72 kg

Table 2 presents the material properties of the concrete specimens tested based on the ASTM standards. All the material properties conformed with the acceptable ranges provided by the ASTM standards.

Table 2. Concrete material properties

Material	Material Property	ASTM Standard	Value
Coarse Aggregates	Moisture Content	ASTM C566	1.09%
Fine Aggregates			3.33%
Coarse Aggregates	Fineness Modulus	ASTM C136	6.46
Fine Aggregates			2.51
Coarse Aggregates	Specific Gravity	ASTM C127	2.78
Fine Aggregates		ASTM C128	2.65
Cement		ASTM C188	3.15
Coarse Aggregates	Absorption	ASTM C127	1.65%
Fine Aggregates		ASTM C128	3.99%
Coarse Aggregates	Unit Weight	ASTM C29	1698.67 kg/m^3
Fine Aggregates			1586.67 kg/m^3

The concrete was mixed using a concrete mixer and transferred to wooden molds as shown in Fig. 1. It was left to dry for 24 hours before being placed in water for curing for 28 days. For this study, three (3) set-ups were conducted: uncoated specimens, seawater-derived $Mg(OH)_2$ coated specimens, and commercially available $Mg(OH)_2$ coated specimens. The study included four (4) replicates for each set-up, resulting in a total of 12 samples.

Fig. 1. Concrete placed in wooden molds

2.2 Seawater Preparation for Magnesium Hydroxide Extraction

Due to the inherent limitations of natural seawater for this study, such as sample heterogeneity and sourcing challenges, synthetic remineralized seawater was chosen as the primary feedstock for the magnesium hydroxide extraction. To determine the amounts required, stoichiometry was performed utilizing a target amount of 0.002 g/mm^2 for each specimen face to ensure a balance between coating thickness and adhesion property. For a 100 mm cubical specimen, this translates to 20 g of $Mg(OH)_2$ slurry per specimen face. Following the recommended ratio of 57.5% total solid weight by Merachtsaki et al. [10], a target amount of 11.30 g of magnesium hydroxide precipitate per face was determined. The relevant chemical equations depicting the relationships between involved reactants are shown in Eq.1 and Eq. 2.

$$CaO + H_2O \rightarrow Ca(OH)_2 \quad \text{(Eq.1)}$$

$$Ca(OH)_2 + Mg^{2+} \rightarrow Mg(OH)_2 \downarrow + Ca^{2+} \quad \text{(Eq.2)}$$

The required amount of seawater was calculated based on the estimated magnesium ion concentration at 1.29 g/L [11]. This yields a required equivalent mass of 4.71g of Mg^{2+} or volume of 3.65 L for each specimen face tested. The seawater batch was then aerated through an ½ inch venturi aerator for 8 minutes.

2.3 Surface Coating Design Materials

In this study, the commercially available magnesium hydroxide was produced in-lab by hydrating magnesium oxide according to the chemical reaction shown in Eq. 3.

$$MgO + H_2O \rightarrow Mg(OH)_2 \quad \text{(Eq.3)}$$

Following extensive agitation and mixing, the resultant product was allowed to oven dry to achieve a powdered form. This solid mass constitutes the commercial portion of the precipitate intended for the coating design.

2.4 Testing Materials

To determine the effectiveness of the seawater-derived $Mg(OH)_2$ coating, the concrete specimens were exposed to 4M sulfuric acid through an improvised testing chamber as shown in Fig. 2. The concrete specimens were manually sprayed for 4 days.

Fig. 2. Improvised testing chamber

Following the exposure to sulfuric acid, the compressive strength of the concrete specimens was tested using a Universal Testing Machine (UTM). To prevent contamination of acid in the machine, specimens were first placed in ice bags.

3. METHODS

The study consists of three main stages namely magnesium hydroxide preparation, surface coating design, and data validation. For this study, the amount of seawater-derived magnesium hydroxide coating designed was for a single face of a 100 mm x 100 mm x 100 mm concrete specimen, where one face was coated with an equivalent of 0.002 g/mm^2 or 20 grams of slurry per specimen.

3.1 Magnesium Hydroxide Recovery from Seawater

Following the preparation of the seawater sample, calcium hydroxide (CaO) or quicklime was separately hydrated using a water-quicklime ratio of 1:4. The required amounts, derived from Equations 1 and 2 to achieve a target of 11.3 g of $Mg(OH)_2$ precipitate, specify a ratio of 10.87 g of CaO per 43.48 g of H_2O per face. The slurry was agitated at a rate of 400 rpm and subsequently subjected to wet sieving through a 100 µm sieve to enhance homogenization and improve the efficiency of the precipitation process. The settlement time required was calculated using Stoke's law in Eq. 4.

$$u = \frac{d^2 g(\rho_s - \rho_f)}{18\eta} \quad \text{(Eq.4)}$$

Where: The diameter (d) of a magnesium hydroxide particle was taken as 6 µm, representative of the lower limit of diameters observed for seawater-derived magnesium hydroxide [12]. The acceleration due to gravity (g) value was set at 9.81 m/s^2 and the dynamic viscosity (η) of water was taken to be that at 25^{o}C with a value of 0.000891 kg/m-s. The densities of the particle (ρ_s) and the surrounding medium (ρ_g) are 2,340 kg/m^3 and 997 kg/m^3, respectively. These inputs result in a settling velocity of 2.96 x 10-5 m/s. For a full 1-L Erlenmeyer flask with an approximate height of 220 mm, a required settlement time of two hours is determined. After two hours, the liquid was decanted, and the remaining mixture was further subjected to dual filtration using filter paper as seen in Fig. 3.

Fig. 3. Filtration of solids

Subsequently, the acquired solids underwent a washing procedure with water to eliminate impurities, followed by a 24-hour oven-drying process to achieve a pure solid state as seen in Fig. 4. The solid mass obtained in this procedure represents the commercial fraction of the precipitate designated for use in the coating design.

Fig. 4. Oven-drying of solids

3.2 Surface Coating Design

3.2.1 Coating Adhesion

Concrete sewer, industrial, or agricultural pipes may be subjected to fast liquid flow, posing a risk of the coating detaching due to the high pressure experienced [13]. To address this issue, adhesives are integrated to strengthen the bond of the coating with the concrete surface.

Previous studies investigated methylcellulose (MC) and carboxymethylcellulose (CMC) as adhesives, testing six concentrations using pull-off bond testing and sewage flow simulation. MC concentrations of 1%, 0.4%, and 0.1% (labeled M1m, M2m, and M3m) were tested, alongside a 4:1 MC to CMC ratio with the same concentrations (labeled M1mc, M2mc, and M3mc). Coating thickness variations aimed to match typical values for cementitious and polymeric coatings, ranging from 0.5 mm to 1.5 mm, with application rates of 0.0018-0.002 g/mm² for thick layers and 0.0008-0.001 g/mm² for thin layers [14].

Results from the pull-off test showed that the 1% concentration of MC (M1m) exhibited the highest bond strength for both thick and thin layers, achieving 0.6 MPa and 0.5 MPa, respectively. The M2m and M1mc coatings

demonstrated the second-highest bond strength, consistently at 0.3 MPa, which was deemed adequate for concrete surface coating [14].

In addition to the pull-off test, an experimental setup was conducted to assess the coating's performance under high velocities. A cylindrical coated specimen was positioned at one end of a tube, where water velocities up to 14 m/s were applied. Both rapid and slow water circulations were tested to evaluate adhesion under varying conditions. Rapid circulation involved a 20-minute immersion under high velocities, while slow circulation maintained a velocity of 0.05 m/s over a one-month period [14].

Comparing the coatings with the highest bond strengths, M1m and M1mc, visual inspection revealed that M1m exhibited better adhesion, whereas M1mc showed significant coating detachment. The results suggest that using MC without the addition of CMC yielded the best performance [14]. Therefore, MC was selected as the coating adhesive in this study to enhance the adhesion of seawater-derived magnesium hydroxide with the concrete specimen.

3.2.2 Coating Slurry

The composition of the coating slurry was adopted from Merachtsaki et al., wherein the recommended ratios comprised 57.5% of solid weights (11.5 g/specimen) and 42.5% deionized water weights (8.5 mL/specimen) [15]. To produce the slurry, the deionized water was heated to 90°C to enhance the solubility and reaction rates of the solutes. Once the temperature stabilized at 90°C, 0.2 grams of MC was added as seen in Fig. 5. This mixture was manually stirred until it cooled to room temperature.

Fig. 5. MC and deionized water mixture

Following this, 11.3 grams of seawater-derived magnesium hydroxide was added under continuous agitation until a homogeneous slurry was achieved, as shown in Fig. 6.

Fig. 6. Seawater-derived magnesium hydroxide protective coating slurry

The coating was placed on the surface of the concrete using a trowel and spatula. Due to the thinness of the coatings, which made manual measurement impractical, it was assumed that the coatings were uniformly applied across all concrete specimens. The coating was left to dry for 1 day before its exposure to sulfuric acid.

3.2.3 Validation

To assess the effectiveness of the seawater-derived magnesium hydroxide protective coating, the compressive strength of the concrete specimens was compared with that of concrete specimens coated with commercially available magnesium hydroxide and uncoated concrete specimens. The applied seawater-derived $Mg(OH)_2$ and commercially available coatings are shown in Fig. 7 and Fig. 8, respectively.

Fig 7. Seawater-derived $Mg(OH)_2$ coating on concrete specimen

Fig. 8. Commercially available $Mg(OH)_2$ coating on concrete specimen

For each set-up, 4 replicates were done. All set-ups were placed in the improvised testing chamber and exposed to 4M sulfuric acid via manual spraying over a period of 4 days. Subsequently, the compressive strength was evaluated using a Universal Testing Machine (UTM) at a load rate of 140 kg/cm^2. The set-up for the compressive strength test is shown in Fig. 9.

Fig. 9. Compressive strength test setup

4. RESULTS AND DISCUSSION

4.1 Compressive Strength Test

Table 3 presents the actual compressive strength results obtained for the control, seawater-derived $Mg(OH)_2$ coated, and commercially available $Mg(OH)_2$ coated concrete specimens while Fig. 10 displays the results in a box-and-whisker plot format.

Table 3. Raw results for compressive strength test

Compressive Strength (MPa)			
Trial	Control	SW $Mg(OH)_2$	CA $Mg(OH)_2$
1	22.25	24.55	26.54
2	21.39	23.97	26.90
3	21.75	24.66	27.56
4	22.19	23.86	26.13
Average	21.89	24.26	26.78

Evaluating the compressive strength results, it was observed from the box-and-whisker plot that the data retrieved from the control, seawater-derived $Mg(OH)_2$, and commercially available $Mg(OH)_2$ all fall within their respective interquartile ranges. This indicates that there are no outliers present within the data sets for these groups, suggesting consistency and reliability in the compressive strength results.

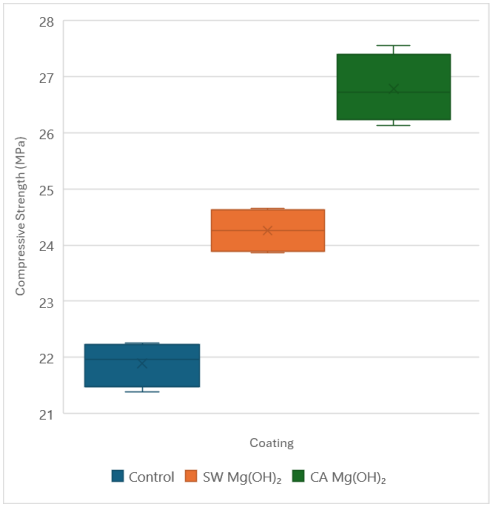

Fig. 10. Compressive strength results in box-and-whisker plot

The average compressive strength of specimens coated with seawater-derived $Mg(OH)_2$ was observed to be 24.26 MPa, while the average compressive strengths for commercially available $Mg(OH)_2$ coated and uncoated concrete specimens were 26.78 MPa and 21.89 MPa, respectively. Comparing these results to the allowable yield compressive strength for structural concrete as per ACI 318-19, set at 24.5 MPa, reveals that the commercial coating achieved a strength value 22.34% higher than the control specimen and 9.31% greater than the acceptable limit. While neither the seawater-based coating nor the uncoated specimens met this acceptance criterion, it may be noted that the seawater-based samples showed a strength value 0.98% below the limit.

The lesser decrease in strength compared to the uncoated specimens can be attributed to the anti-corrosive properties inherent in magnesium hydroxide. An alkaline-based concrete surface coating can offer resistance to sulfuric acid by either maintaining surface alkalinity or actively neutralizing sulfuric acid upon contact. The mechanism is explained by the chemical equation shown in Eq. 5.

$$Mg(OH)_2 + H_2SO_4 \rightarrow MgSO_4 + 2H_2O \quad \text{(Eq.5)}$$

The magnesium hydroxide-based coating acts as a protective shield by initially neutralizing sulfuric acid through this chemical process, thereby preventing it from affecting the underlying concrete surface beneath the coating. Serving as an alkaline reservoir, magnesium hydroxide creates a buffer that inhibits the growth of acidophilic bacteria and enhances the concrete's resistance to shifts in its naturally alkaline pH. This dual action not only safeguards the structural integrity of sewer pipes, but also mitigates the formation of acidic conditions that could lead to the deterioration of the concrete substrate.

On the other hand, the drastic decrease in the compressive strength of the uncoated specimens can be attributed to the consumption of calcium hydroxide ($Ca(OH)_2$) and calcium silicate hydrates (CSH gel). These two components are present in Portland cement, a material commonly used in the production of concrete pipes. They play a crucial role in contributing to the durability, versatility, and alkalinity of concrete [16]. However, when concrete is exposed to acidic environments, such as those found in sewage systems, the pH levels of the concrete decrease. This acidic exposure leads to the degradation of $Ca(OH)_2$ and CSH gel. As these components break down, the binding strength of the cement is compromised. The degradation process also results in the formation of gypsum, which further exacerbates the deterioration by causing cracks and shrinkage within the concrete structure. This combination of factors ultimately leads to significant deterioration of the concrete. The integrity and performance of the concrete are severely impacted, making it less durable and more prone to damage under load and environmental stresses.

The superior performance of the commercially available $Mg(OH)_2$ coated specimens over the seawater-derived $Mg(OH)_2$ coated specimens are inherently attributed to the amount of $Mg(OH)_2$ ions present in the coating mixes. While the commercially sourced mix consisted solely of pure $Mg(OH)_2$ ions, the seawater-derived mixture contained additional ions of sodium (Na^+), magnesium

(Mg^{2+}), calcium (Ca^{+}), sulfate (SO_4^{2-}), bicarbonate (HCO_3^{-}), and chloride (Cl^{-}). These additional ions were produced from intermediate processes during the extraction of the precipitate. As the capability of the produced coating material was reliant on the contact-based neutralization reaction that occurred between the induced sulfuric acid (H_2SO_4) and $Mg(OH)_2$, the presence of additional ions may have possibly interceded this process, resulting in the perceived reduced effectiveness in the seawater-derived coatings [17]. Moreover, the extraction and design of coatings were conducted in a laboratory primarily intended to test the mechanical properties of concrete. From this, general impurities stemming from the environment may have introduced experimental errors to the production processes involved in the methodology of this study, also contributing to the reduced effectiveness of seawater-based coatings.

In addition, as observed in Equation 5, the reaction between one mole of $Mg(OH)_2$ and H_2SO_4 yields one mole of magnesium sulfate ($MgSO_4$) and two moles of water. Magnesium sulfate, the resulting compound, is a soluble salt that prevents the build-up of solid obstructions in sewer pipes by dissolving readily in water. This naturally occurring compound poses no environmental hazards, as it disperses harmlessly into wastewater treatment facilities or natural water bodies, causing minimal environmental impact. This demonstrates that $Mg(OH)_2$ and its subsequent reaction with sulfuric acid have no detrimental side effects.

From this test, the findings suggest a potential for exploring various ratios and combinations to optimize the coating mixture. Although the seawater-based coating is less effective than the commercial magnesium hydroxide-based coating, it still shows notable enhancement over the control specimen. This highlights promising prospects in corrosion mitigation and underscores the potential for further enhancing performance through refinement of the mixture. By standardizing factors such as coating concentration, application methods, and the incorporation of additional additives, there is a possibility to improve various mechanical properties of the concrete.

5. CONCLUSION

The prevalence of SRB in the anaerobic environment of concrete systems results in the production of hydrogen sulfide, making such infrastructure vulnerable to hydrogen sulfide-induced corrosion. While current mitigation methods are effective, their continual application has detrimental effects on the environment.

As such, this study extracted magnesium hydroxide from seawater for use in the production of protective coatings against hydrogen sulfide-induced corrosion in concrete structures as a sustainable alternative approach. Three distinct set-ups were tested: uncoated concrete specimens, seawater-derived $Mg(OH)_2$ coated concrete specimens, and commercially available $Mg(OH)_2$ coated concrete specimens. Four replicates were produced for each set-up, with specimens exposed to 4M sulfuric acid in an improvised testing chamber for a four-day period. After the exposure period, the compressive strength of each specimen was tested to determine the effectiveness of the coatings.

Results showed that coatings made with commercially available magnesium hydroxide still performed better than those made with seawater-derived magnesium hydroxide. Nevertheless, specimens coated with seawater-derived magnesium hydroxide exhibited higher compressive strengths than uncoated specimens, indicating the effectiveness of the protective coating in mitigating the corrosive effects of sulfuric acid.

Thus, the study highlights the potential of using seawater-derived magnesium hydroxide to mitigate hydrogen sulfide-induced corrosion in concrete systems. To further improve the effectiveness of seawater-derived magnesium hydroxide, future studies should explore the production of coatings with varying proportions of commercially available and seawater-derived magnesium hydroxide to determine if a mixture of both variants would be more effective in mitigating hydrogen sulfide-induced corrosion in concrete systems. Moreover, given the short time frame of the study, an accelerated spraying procedure using 4M sulfuric acid over four days was employed. Future studies are recommended to explore longer testing periods using a 0.2M sulfuric acid concentration to better replicate the ambient and anaerobic environments of actual concrete systems vulnerable to hydrogen sulfide-induced corrosion. Finally, this study primarily used a manual spraying method to expose specimens to sulfuric acid. Future studies should investigate whether automated spraying methods would yield comparable or different results using the same configurations.

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