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Influence of sulfate ion and associated cation type on steel reinforcement corrosion in concrete powder aqueous solution in the presence of chloride ions

Fouzia Shaheen, Bulu Pradhan *

Department of Civil Engineering, Indian Institute of Technology Guwahati, Guwahati 781039, India

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ABSTRACT

State of rebar corrosion in concrete powder aqueous solution contaminated with chloride and sulfate ions has been determined by conducting a potentiodynamic polarization test. XRD analysis and FTIR spectroscopy were also performed. From the results, different zones of corrosion in terms of potential ranges have been identified. The presence of Na_2SO_4 has mitigated the effect of chloride ions whereas the presence of MgSO_4 has stimulated the effect of chloride ions on reducing the passivity of steel reinforcement in a chloride environment. Ordinary Portland cement performed better against Mg-oriented sulfate attack whereas Portland pozzolana cement performed better against Na-oriented sulfate attack in the presence of chloride ions.

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1. Introduction

Concrete is a heterogeneous material with many special characteristics, including high alkalinity of the pore solution, high electrical resistivity and has a structure that acts as a physical barrier for mass transport and crack behaviour [1]. Concrete durability problems are the major cause of concern all over the world especially in the coastal areas where the structures are undergoing deterioration well before their expected life and need proper attention and care [2]. In the marine environment, chloride and sulfate salts do exist concomitantly. The conjoint presence of these salts may cause deterioration of concrete due to reinforcement corrosion and sulfate attack [3].

Chloride ions enter into concrete through two sources such as internal chloride (at the time of preparation of concrete, through chloride contaminated aggregates, chloride-containing admixtures, or mixing water) and external chloride (entering from the external environment into the hardened concrete through deicing salts, sea water, soil and ground water) [4]. In the hardened cement paste, when both chlorides and sulfates exist conjointly, they react with tricalcium aluminate (C_3A) to form various compounds. Chloride ions react with C_3A to form calcium chloroaluminate (Friedel's salt) [5]. Sulfate ions react with calcium hydroxide and hydrated C_3A to form gypsum and ettringite respectively, which lead to expansion and disruption of hardened concrete. The higher proportions of C_3A would reduce the level of

reinforcement corrosion by lowering corrosion-inducing free chlorides from the concrete pore solution, but it would pose a serious concrete durability problem in terms of sulfate attack [6]. The electrolytic pore solution of concrete has a pH value between 12.5 and 13.5 due to the presence of calcium hydroxide along with small amounts of Na_2O and K_2O [1]. In this highly alkaline environment, a thin protective layer known as a passive layer of $\gamma\text{-Fe}_2\text{O}_3$ is formed on the rebar surface [7–9]. However the free chloride content (also known as water soluble chloride) in the pore solution of concrete breaks down the passive layer of steel and initiates corrosion [10]. Sulfate ions enter into concrete externally from exposure condition and internally through aggregates, mixing and curing water, and admixtures [3]. Sulfate ions can be associated with magnesium, calcium and sodium cations. Since the solubility of calcium sulfate is very low, magnesium and sodium sulfate are mainly responsible for the deterioration of concrete due to sulfate attack [11].

In the conjoint presence of chloride and sulfate ions, the mechanism of deterioration of concrete becomes complex due to the simultaneous interaction of these ions with hydrated cement phases. Further, the cation type associated with these aggressive ions makes the mechanism even more complex. From the review of literature, it is inferred that different researchers have studied the corrosion behaviour of steel embedded in concrete exposed to chloride, sulfate and mixed chloride-sulfate environment. Zuquan et al. [12] reported that the presence of sulfate ions in a composite solution enhanced the resistance to chloride penetration into the concrete at the early stage of exposure, but the opposite behaviour was observed at a later exposure period. Further from XRD results, the authors observed the formation of a higher amount of

* Corresponding author.

E-mail address: bulu@iitg.ernet.in (B. Pradhan).

Nomenclature

CCA	Calcium chloroaluminate
E	Ettringite
G	Gypsum
CH	Calcium hydroxide
MH	Magnesium hydroxide
CC	Calcium carbonate
T	Thaumasite
Q	Quartz

gypsum and ettringite in the concrete exposed to sulfate solution as compared to that exposed to composite sulfate-chloride solution. A study conducted by Al-Amoudi and Maslehuddin [7] on the respective effect of chloride and sulfate ions on corrosion of steel in cement paste indicated that sulfate ions are hardly able to initiate reinforcement corrosion. However considerable reinforcement corrosion was observed in specimens immersed in mixed chloride-sulfate solution. Jarrah et al. [13] reported that the time to initiation of reinforcement corrosion was higher in blended cement than plain cement in all chloride and chloride-sulfate solutions and the sulfate ions alone do not initiate the reinforcement corrosion but they increase the corrosion activity once the corrosion was initiated due to the conjoint presence of chloride and sulfate salts. Dehwah et al. [14] have found that the presence of sulfate ions in a chloride environment did not affect the time to initiation of reinforcement corrosion but corrosion current density increased with an increase in the concentration of sodium sulfate and magnesium sulfate.

The presence of chloride and sulfate salts significantly affects the pore solution chemistry of concrete. The pore solution of concrete significantly varies in the concentration of hydroxyl ions, sulfate ions, chloride ions, solubility of metal cations and temperature [15]. Different researchers have investigated the corrosion behaviour of reinforcing steel in simulated concrete pore solutions. Dehwah et al. [9] studied the effect of chloride and sulfate contamination on the pore solution chemistry in plain and blended cements and the results from the study indicated that the chloride binding capacity of both plain and blended cements decreased due to the concomitant presence of chloride and sulfate salts. It was also observed that the increase in chloride and sulfate ions in mixed chloride-sulfate solution enhances the corrosion of steel reinforcement. Zhang et al. [16] have found that the passivity breakdown potential or pitting potential is lower with higher chloride concentrations and they also observed that the higher pH of the simulated concrete pore solution facilitates the passivation of steel, whereas the higher concentration of Cl^- leads to a passivity breakdown at relatively lower potential. Ghods et al. [17] have investigated the growth of oxide film on steel surface in saturated calcium hydroxide solution with different amounts of NaOH, KOH and $\text{Ca}(\text{SO}_4)_2$. The authors reported that pore solution composition has an effect on the protective properties of passive oxide film and the presence of SO_4^{2-} ions in the pore solution has a significant negative effect on the protective properties of the passive oxide film. Chen et al. [18] reported that in chloride free saturated calcium hydroxide solution, steel remains in a passive state and the corrosion current of steel was very low. On the other hand in the saturated calcium hydroxide with 0.5 M NaCl, the corrosion current density increased considerably and the steel surface was unstable with chloride attack and localized corrosion appeared with FeCO_3 and Fe_2O_3 as main corrosion products on its surface. Padilla and Alfantazi [19] found that the most severe corrosion damage in terms of corrosion rate and degradation of the corrosion film formed on the surface and was observed when galvanized steel was immersed in $\text{NaCl} + \text{Na}_2\text{SO}_4$ solution whereas the best performance was observed in $\text{MgCl}_2 + \text{Na}_2\text{SO}_4$ solution. Pradhan and Bhattacharjee [20] have

conducted a potentiostatic study on reinforcing steel in chloride contaminated concrete powder solution extracts. The authors reported that chloride content has the strongest influence in governing zones of corrosion of steel reinforcement as compared to other parameters. Aal et al. [21] have reported that the pitting corrosion current density in constant $\text{Ca}(\text{OH})_2$ solution increased with the increase in SO_4^{2-} and Cl^- ions, whereas under the constant concentrations of SO_4^{2-} and Cl^- ions, pitting corrosion current density decreased with an increase in the concentration of $\text{Ca}(\text{OH})_2$.

From the review of past research work, it is observed that very few studies have been conducted to evaluate the corrosion performance of steel reinforcement in simulated concrete pore solutions contaminated with composite chloride-sulfate salts. Further, in these studies mostly saturated calcium hydroxide solution has been taken as the simulated pore solution. On the other hand, concrete powder aqueous solutions can also be taken as the electrolytic concrete pore solution. Since concrete powder from which the aqueous solution is extracted, is a mixture of cement hydrates, coarse aggregates and fine aggregates, the aqueous solution may represent the electrolytic pore solution of concrete more closely as compared to saturated calcium hydroxide solution. From the literature review, it is inferred that the work on corrosion performance of steel in concrete powder aqueous solutions contaminated with chloride ions and/or composite chloride-sulfate ions is meager. Therefore, in the present research work, an attempt has been made to study the electrochemical behaviour of steel in electrolytic concrete powder aqueous solutions contaminated with chloride and composite chloride-sulfate ions through anodic polarization curves by conducting a potentiodynamic polarization test. Further to analyze the effect of chemical composition of the concrete powder aqueous solutions on corrosion behaviour of steel reinforcement, ionic concentration, pH and conductivity of concrete powder aqueous solutions were determined. In addition, for the purpose of analyzing the changes in phase composition of hardened concrete and for identifying different functional groups associated with different products formed in concrete in the presence of chloride ion and sulfate ions, X-ray diffraction (XRD) analysis and Fourier transform infrared (FTIR) spectroscopy were conducted.

2. Experimental work

The experimental program has been designed to evaluate the corrosion performance of steel in ordinary Portland cement (OPC) and Portland pozzolana cement (PPC) concrete powder aqueous solutions contaminated with different concentrations of chloride ions and sulfate ions. The details of materials used, test specimens and tests conducted in the present investigation are presented below.

2.1. Materials used and specimen preparation

Concrete cube specimens with a size of 150 mm were prepared using ordinary Portland cement satisfying IS: 12269-1987 [22] and ASTM Type I [23]; and Portland pozzolana cement satisfying IS: 1489-1991 [24] and ASTM Type IP [25] with a water-cement ratio (w/c) of 0.50. The chemical composition of OPC and PPC determined by XRF (X-ray fluorescence) analysis are presented in Table 1. Locally available river sand was used as fine aggregate. The specific gravity of sand is 2.61 and it is conforming to grading zone II as per IS: 383-1970 [26] and as per ASTM C33/C33M-13 [27]. The coarse aggregate of quartzite origin

Table 1

Chemical composition of cement determined by X-ray fluorescence (XRF) analysis.

Compound (wt.%)	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃	LOI
Ordinary Portland cement (OPC)	65.2	19.2	5.2	2.4	3.4	0.3	0.62	1.5	1.4
Portland pozzolana cement (PPC)	64.7	20.52	4.2	3.4	1.55	0.35	1.31	1.6	1.2

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