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ORIGINAL PAPER

Role of alkaline nitrites in the corrosion performance of steel in composite cements

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Abstract The influence of alkaline nitrites on the inhibition of corrosion of steel in binary and ternary cement environments was tested. pH measurements carried out for binary and ternary cement extracts showed that the alkalinity of the cement was not affected by making use of binary and ternary cements. Gravimetric measurements showed that the decrease in the corrosion rate of steel in different systems follows the order: Ternary > (OPC + PSC) > (OPC + PPC) > (PPC + PSC). Potential–time studies indicated that the ability to maintain the passivity of steel in different systems also follows the order as above. Potentiodynamic polarisation studies for steel in binary and ternary cement environments showed the favourable influence of the presence of higher amounts of chlorides. Nitrites of sodium, potassium and calcium act as anodic inhibitors and they compete with chloride ions for the ferrous ions at the steel to form a film of ferric oxide. An efficiency as high as 91% is obtained for the ternary system containing 1% chloride and 0.5% nitrite. The degree of surface coverage showed a maximum value for the ternary system (>0.9) even in the presence of a higher amount of chloride thereby indicating the better performance of the system.

Keywords Composite cements · Nitrites · Corrosion · Concrete · Inhibitors

1 Introduction

Steel embedded in concrete is normally in a passive state, owing to the formation of a thin layer of iron oxide on its surface, which remains stable even in a highly alkaline environment of the concrete [1]. The embedded steel starts to corrode when the destruction of this protective film occurs owing to the corrosive attack of chloride ions on steel and also due to the carbonation of concrete cover by its reaction with carbon dioxide. This causes a reduction in the alkalinity of the concrete. The use of corrosion inhibitors may be a better alternative than other protective methods, because of its ease of application and lesser cost [2–7]. Corrosion inhibitors have been used successfully for many decades in pipelines and tanks etc. However their use in concrete is very scarce and very much limited [8–13]. The effectiveness of $NaNO_2$ as an inhibitor for the corrosion of steel in autoclaved products has been investigated as a counter measure for the decrease in alkalinity. Soviet Union has pioneered the use of calcium nitrite as a protective strategy against corrosion caused by de-icing salts. Calcium nitrite has been used as an inhibitor for several years in Japan and United States. The Japanese have made extensive use of sea sand in concrete in 1970s. The first patent for the commercial application of nitrite based inhibitor was awarded to a company in 1977. The competitive role played by an inhibitor and aggressive ions on their adsorption on mild steel surfaces has been studied [14]. However systematic and detailed studies on the influence of alkaline nitrites such as sodium, potassium and calcium inhibitors on the corrosion of steel in composite cement have not yet been carried out. In the present study, the inhibition performance of different nitrites on the corrosion of steel in composite cement systems has been investigated using different electrochemical and non-electrochemical techniques.

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2 Experimental details

2.1 Materials used

Ordinary Portland cement (OPC) [IS:8112-1989], Portland pozzalona cement (PPC) [IS: 1489 (part-1) 1991-fly ash based] and Portland slag cement (PSC) [IS: 455-1989] were used throughout the study. The chemical composition of OPC, PPC and PSC are given in Table 1.

2.2 Chemicals used

$NaNO_2$ (AR) and KNO_2 (AR) procured from Reidel Research Laboratory chemicals, India and $Ca(NO_2)_2$ imported from Russia were used as such, without any further purification.

2.3 Preparation of cement extracts

Cement was sieved through 90-µm sieve and extracts were prepared as follows: To 100 g of cement, 100 ml of distilled water was added and shaken vigorously using a Microid flask mechanical shaker for about 1 h. The extracts were then collected by filtration.

2.4 Composite systems

Binary systems such as (OPC + PSC), (OPC + PPC), (PPC + PSC) and a ternary system namely (OPC + PPC + PSC) were used in the present study. The flow chart for making composite cements is given in Fig. 1. Proportionate weights of individual components were taken and then mixed thoroughly using a roller mixer. Extracts of composite systems were then prepared as above.

2.5 Methods

2.5.1 *pH measurements*

A portable Roy instrument (Model pH 300) pH meter, already calibrated using a buffer solution of pH 9, was used to measure pH values of both binary and ternary cement extracts. Measurements of pH values were done initially and also at the end of an exposure period of 6 months.

Table 1 Composition of OPC, PPC and PSC cements

Constituent	wt.%		
	OPC	PPC	PSC
SiO_2	20–21	28–32	26–30
Al_2O_3	5.2–5.6	5.0–8.0	9.0–11.0
Fe_2O_3	4.4–4.8	4.9–6.0	2.5–3.0
CaO	62–63	43–45	44–46
MgO	0.5–0.7	1.0–2.0	3.5–4.0
SO_3	2.4–2.8	2.4–2.8	2.0–2.4
LOI	1.5–2.5	3.0–3.5	1.5–2.5

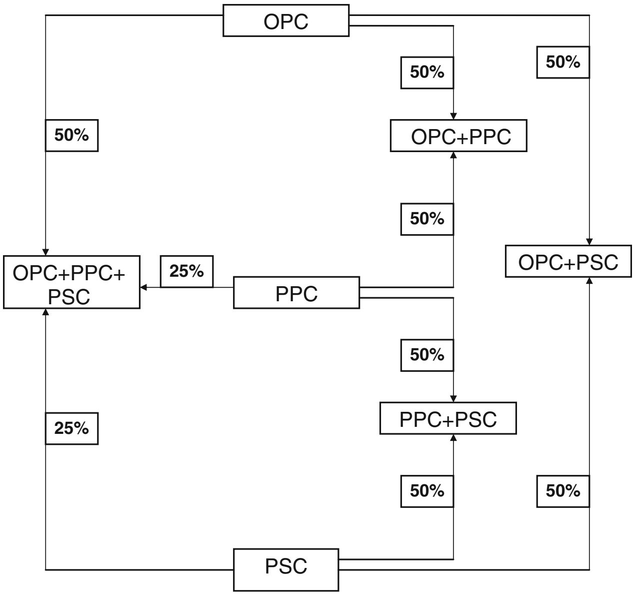

Fig. 1 Flow chart for the preparation of binary and ternary cements

2.5.2 *Weight loss measurements*

Thermo mechanically treated (TMT) steel rods of size 12 mm diameter and 60 mm length were used. These rods were mechanically polished to remove any rust on the surface, subsequently polished using different grades of emery papers and then degreased with acetone. Initially weighed TMT bars in triplicate were suspended in a PVC see-through cell containing 250 ml of different cement extracts and cement extracts containing different concentrations of chlorides and different nitrites. The test solution was changed every week in order to induce accelerated corrosion. The temperature was maintained constant at 35 ± 1°C. Rods were weighed after an exposure period of 6 months and weight losses were noted. The corrosion rate was calculated using the equation [15].

$$\text{Corrosion rate (mmpy)} = 87.6\ W/DAT \qquad (1)$$

where W is the weight loss in milligrams; D the density of the material in g cm^{-3}; T the time duration in hours; and A is the area of the specimen in cm^2.

2.5.3 *Open circuit potential measurements*

Half cell potentials of steel in different cement extracts were periodically monitored with reference to a saturated calomel electrode (SCE) using a voltmeter of high input impedance of the order of 10 MΩ. The potential of steel in different extracts was monitored at regular intervals of time for an exposure period of 6 months.

2.5.4 *Potentiodynamic polarisation studies*

Potentiodynamic polarisation studies were carried out using a TMT steel rebar of exposed area 1 cm^2 as the working electrode, a saturated calomel electrode as the reference electrode and a platinum foil of 3 cm × 3 cm area as the counter electrode. The working electrode was mechanically polished first, followed by polishing successively with emery paper of 1/0, 2/0, 3/0 and 4/0 grade and then degreased with acetone. Potentiodynamic polarisation studies were carried out using steel in binary and ternary cement extracts and extracts containing different percentages of chlorides using an ACM instrument (UK). This instrument has provisions for programs to evaluate corrosion kinetic parameters such as I_{corr}, E_{corr}, b_a and b_c. The potentiodynamic condition corresponds to a potential sweep rate of 1 mV s^{-1} and a potential range of $+200$ to -200 mV from OCP. Polarisation studies were also carried out for all the systems containing 0.5% $NaNO_2$. All the studies were carried out at a room temperature of 35 ± 1°C.

2.5.5 *A.C. impedance measurements*

Impedance measurements were carried out using steel in binary and ternary cement extracts and extracts containing different percentages of chlorides using an ACM Instrument (UK). Impedance measurements were also carried out for all the systems containing 0.5% $NaNO_2$. Impedance parameters were evaluated for steel in binary and ternary cement extracts as described elsewhere [14].

2.5.6 *Visual observations*

During weight loss measurements rebar specimens were taken out daily and carefully examined for the formation of any corrosion product, such as red rust, on its surface using a magnifying lens. The time taken for the visible formation of red rust was noted for all the systems studied. At the end of the exposure period, specimens were removed from the test solutions, washed well with water and examined using a microscope.

3 Results and discussion

3.1 pH measurements

Values of pH for different systems in the presence and absence of nitrites containing different percentages of chlorides were measured. Initial pH values for different binary systems such as (OPC + PSC), (OPC + PPC) and (PPC + PSC) were found to be 12.50, 12.27 and 12.01 respectively. This shows that the alkalinity is not affected while making different binary systems by combining different cements [16]. The initial pH value for ternary system was 12.52. After adding chlorides and nitrites there was no any appreciable change in the initial pH values for both binary and ternary systems. After the exposure period of 6 months, the final pH value for plain extracts (both binary and ternary) was 10.5. Inhibited binary systems showed pH values in the range 11–11.5 at the end of the exposure. On the other hand, the ternary system showed a pH value of 12 by maintaining the alkalinity near the steel surface even after 6 months of exposure. This may be due to the fact that plain extract readily reacts with the atmosphere and becomes carbonated. However the extent of reaction of the inhibited system with the atmosphere is very slow.

3.2 Visual observation

All the systems are visually examined every day for any rust formed on the steel surface. The binary system (OPC + PPC) showed red rust at the end of the first month of exposure. The (OPC + PSC) system showed red rust at the end of the third month's exposure and the (PPC + PSC) system showed red rust within a month of exposure. On the other hand, no rust formation was observed for the ternary system up to an exposure period of 6 months, indicating their better corrosion resistance.

3.3 Corrosion rate by weight loss measurements

Corrosion rate for steel in binary and ternary systems containing different concentration of chloride ions and 0.5% of nitrites of sodium, potassium and calcium are given in Table 2.

The corrosion rate of steel in the binary system namely (OPC + PSC) value is lesser than that in (OPC + PPC) and (PPC + PSC). Steel in the ternary system showed the least corrosion rate. It was also found that incorporation of 0.5% alkaline nitrite in different binary systems drastically brings down the corrosion rate. The efficiency of alkaline nitrites to reduce the corrosion rate of steel in binary system follows the order: $NaNO_2 > KNO_2 > Ca(NO_2)_2$. A similar observation can be made in the case of corrosion rate of steel in the ternary system also.

The difference in the performance of these nitrites can be due to the difference in the extent of solubility of these compounds in water. Sodium and potassium nitrites ionise completely in water. But calcium nitrite ionises comparatively to a lesser extent than other nitrites. So the availability of nitrite ions for reaction on steel is more for

Table 2 Average corrosion rates for steel in binary and ternary systems in the presence of different nitrites (0.5%) and different concentrations of chlorides obtained from weight loss measurements

S.No.	System	Corrosion rate (mmpy)			
		(OPC + PPC)	(OPC + PSC)	(PPC + PSC)	Ternary
1	Plain	0.0679	0.0564	0.1003	0.0556
2	Plain + 0.5% $NaNO_2$ + 0% NaCl	0.0283	0.0226	0.0370	0.0063
3	Plain + 0.5% $NaNO_2$ + 1% NaCl	0.0377	0.0336	0.0619	0.0077
4	Plain + 0.5% $NaNO_2$ + 2% NaCl	0.0708	0.0366	0.0744	0.0288
5	Plain + 0.5% $NaNO_2$ + 3% NaCl	0.0962	0.0460	0.0990	0.0362
6	Plain + 0.5% KNO_2 + 0% NaCl	0.0297	0.0242	0.0393	0.0069
7	Plain + 0.5% KNO_2 + 1% NaCl	0.0385	0.0355	0.0622	0.0085
8	Plain + 0.5% KNO_2 + 2% NaCl	0.0731	0.0452	0.0748	0.0291
9	Plain + 0.5% KNO_2 + 3% NaCl	0.1006	0.0465	0.1113	0.0376
10	Plain + 0.5% $Ca(NO_2)_2$ + 0% NaCl	0.0299	0.0251	0.0397	0.0097
11	Plain + 0.5% $Ca(NO_2)_2$ + 1% NaCl	0.0393	0.0358	0.0683	0.0123
12	Plain + 0.5% $Ca(NO_2)_2$ + 2% NaCl	0.0947	0.0463	0.0814	0.0227
13	Plain + 0.5% $Ca(NO_2)_2$ + 3% NaCl	0.1052	0.0471	0.1857	0.0392

those compounds than for calcium nitrite. The better performance of sodium nitrite can be explained due to the difference in the values of hydrolysis constants for these compounds [17]. Hydrolysis constant for sodium cation in sodium nitrite (14.48 kJ mol^{-1}) is more than that for potassium ion in potassium nitrite (13.0 kJ mol^{-1}) and calcium ion in calcium nitrite (12.70 kJ mol^{-1}). The greater hydrolysis constant of sodium nitrite in water enables it to perform better than other nitrites. Moreover the value of electron affinity is more for sodium (52.87 kJ mol^{-1}) than for potassium (48.38 kJ mol^{-1}) and calcium (0 kJ mol^{-1}).

Nitrites of sodium, potassium and calcium act as "anodic inhibitors" and they compete with chloride ions for sites on the steel surface to form a film of ferric oxide as follows:

$$2Fe^{++} + 2OH^- + 2NO_2^- \rightarrow 2NO + Fe_2O_3 + H_2O \qquad (2)$$

$$Fe^{++} + OH^- + NO_2^- \rightarrow NO + \gamma FeOOH + H_2O. \qquad (3)$$

The addition of sodium chloride to binary and ternary systems containing different nitrites (0.5%) increases the corrosion rate of steel in all the systems.

It was found that an increase in the concentrations of NaCl increases the corrosion rate of steel in both binary and ternary systems. All the nitrites are found to be more effective in reducing the corrosion rate in the (OPC + PSC) system. They are found to perform better in the ternary system. It is also found that the effectiveness of different alkaline nitrites in reducing corrosion decreases with increase in the concentration of NaCl.

3.4 Potential–time studies

Figures 2–5 show the potential–time curves for steel in binary and the ternary systems containing 0.5% $NaNO_2$ and different concentrations of NaCl. It can be seen from Fig. 2 that the OCP of steel in (OPC + PPC) extract shifts to more negative values. The shift in potential is found to be less for the duration of 1 month, when the steel is in a passive state. The potential then shifts to very high negative values and the formation of red rust is observed at the end of the exposure period.

It can be seen from Fig. 3 that steel in the (OPC + PSC) system shows a potential less than −275 mV versus SCE

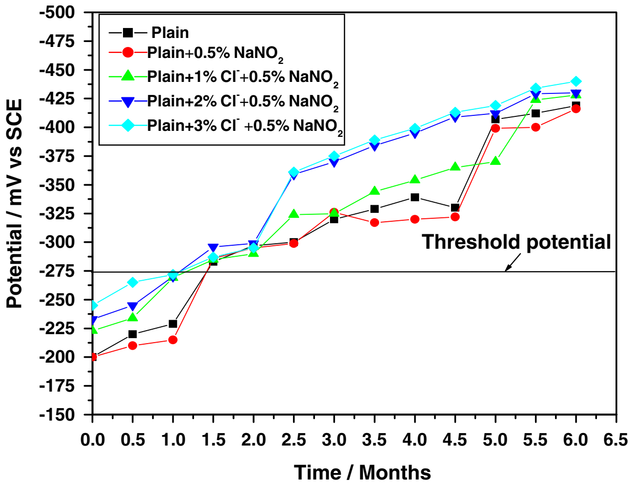

Fig. 2 Potential–time curves for steel in (OPC + PPC) extract and extracts containing different concentrations of chloride and 0.5% $NaNO_2$

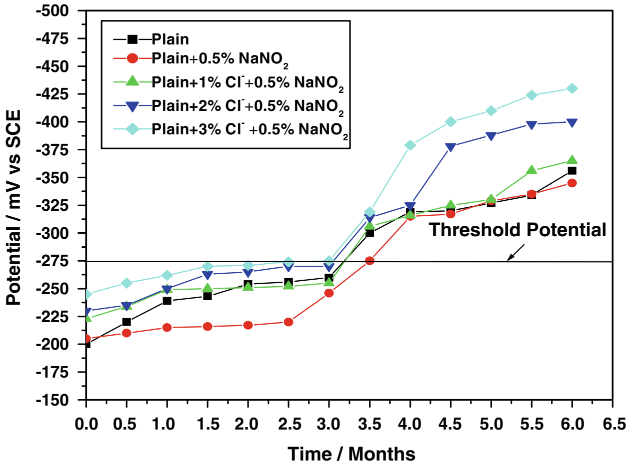

Fig. 3 Potential–time curves for steel in (OPC + PSC) extract and extracts containing different concentrations of chloride and 0.5% $NaNO_2$

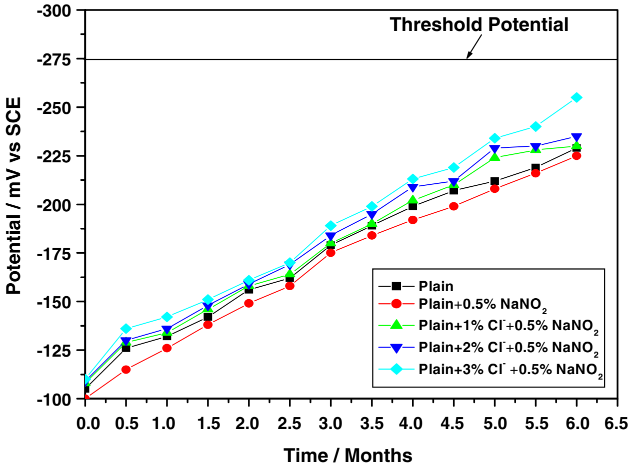

Fig. 5 Potential–time curves for steel in ternary extract and extracts containing different concentrations of chloride and 0.5% $NaNO_2$

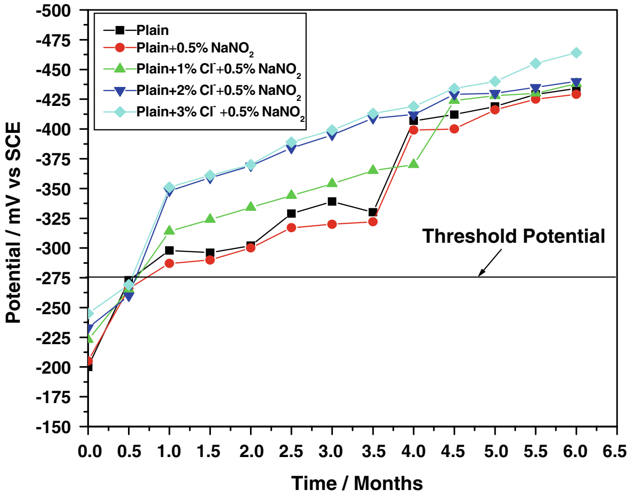

Fig. 4 Potential–time curves for steel in (PPC + PSC) extract and extracts containing different concentrations of chloride and 0.5% $NaNO_2$

up to a duration of 3 months for all the systems, thereby indicating that steel is in a passive condition for a long time. The potential then becomes more negative. The system with chloride shifts the potential to more negative values at the end of 6 months. Figure 4 shows the potential–time curve for steel in (PPC + PSC) extract containing 0.5% $NaNO_2$ and different concentrations of chloride. Steel in this system is found to be in an active condition, even within a month of exposure and exhibited the formation of more rust. It can be seen from Fig. 5 that steel in the ternary system containing 0.5% $NaNO_2$ and different concentrations of chloride remains passive till the end of the exposure period. Unlike other systems, potentials measured are found to be less than the threshold potential for all the systems for an exposure period of 6 months.

Potential–time studies clearly bring out the fact that the ability of different systems to maintain the passivity of steel follows the order: Ternary > (OPC + PSC) > (OPC + PPC) > (PPC + PSC).

Steel in the ternary system shows good performance even in the presence of 3% chloride, because of the fact that a good passive condition around it is maintained throughout. As hydroxide ions present in the cement acts as an inhibitor, the incorporation of an inhibitor such as $NaNO_2$ results in the presence of a mixed inhibitor in the system. The addition of chloride ions to the system may induce corrosion of steel in concrete because they compete with hydroxide ions for defective sites on the steel surface. This observation has been confirmed in the present study.

3.5 Potentiodynamic polarisation studies

Corrosion kinetic parameters for the binary and the ternary systems were evaluated from potentiodynamic polarisation curves for steel in different systems. The binary system (OPC + PSC) shows a lesser corrosion rate of 0.0027 mmpy than the (PPC + PSC) system with a corrosion rate of 0.0035 mmpy. Incorporation of different concentration of chloride ion enhances the corrosion rate to different extents. But the addition of 0.5% $NaNO_2$ reduces the corrosion rate. Sodium nitrite is found to be more effective in the (OPC + PSC) system followed by (PPC + PSC) and (OPC + PPC) systems in decreasing order. These observations are in keeping with those in the weight loss measurements. So among the three binary systems, steel in (OPC + PSC) shows better performance even in the presence of a corrosion accelerator (NaCl) and a corrosion inhibitor ($NaNO_2$).

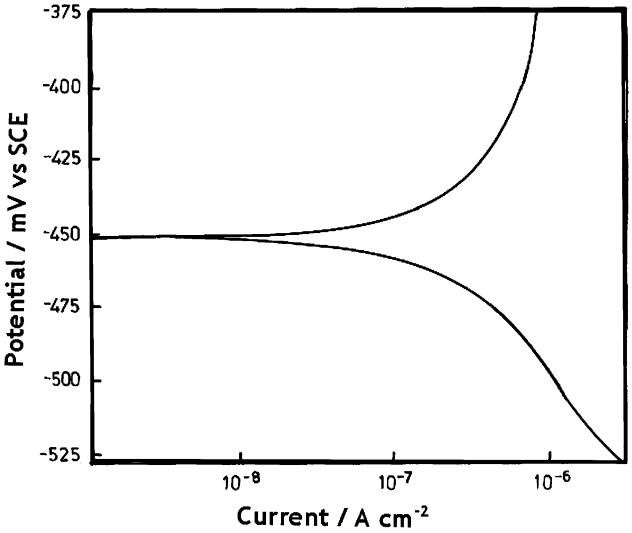

Fig. 6 Potentiodynamic polarisation curve for steel in ternary cement extract

Figure 6 shows the potentiodynamic polarisation curve for steel in ternary cement extract. Table 3 gives corrosion kinetic parameters for steel in the ternary system containing 0.5% $NaNO_2$ and different concentrations of NaCl. E_{corr} for steel shifts to more negative values with increase in the concentration of chloride ions. But incorporation of $NaNO_2$ in the system shifts E_{corr} to less negative values, thereby showing that it acts as an anodic inhibitor.

The anodic Tafel slope for the corrosion of steel in concrete is found to vary from 20 to 91 mV. The cathodic Tafel slope varies from 80 to 230 mV. These limits represent the typical anodic and cathodic slopes for reinforced concrete structures [18].

3.6 A.C. impedance measurements

Impedance diagrams for steel in both binary and ternary systems in the presence of different concentrations of NaCl and 0.5% $NaNO_2$ were obtained and impedance parameters such as charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) were evaluated. The (OPC + PSC) system give R_{ct} values (4.292 × 10^3 Ω cm^2) followed by (OPC + PPC) (3.295 × 10^3 Ω cm^2) and (PPC + PSC) (2.770 × 10^3 Ω cm^2) systems in the decreasing order. The ternary system gives a higher value of R_{ct} (5.103 × 10^3 Ω cm^2). Incorporation of different concentrations of chloride ions decreases R_{ct} values to different extents depending on the concentration. Similarly the addition 0.5% $NaNO_2$ to the system enhances R_{ct} values for different binary systems to different extents. Maximum enhancement is shown by the (OPC + PSC) system followed by the (OPC + PPC) and (PPC + PSC) systems in decreasing order. Double layer capacitance (C_{dl}) values evaluated for different binary systems follow the order:

$$\underset{(9.119\times10^{-5}\,\mathrm{F\,cm^{-2}})}{(\mathrm{PPC+PSC})} > \underset{(8.78\times10^{-5}\,\mathrm{F\,cm^{-2}})}{(\mathrm{OPC+PPC})} > \underset{(7.76\times10^{-5}\,\mathrm{F\,cm^{-2}})}{(\mathrm{OPC+PSC})}$$

The ternary system gives a lesser value of 6.102 × 10^{-5} F cm^{-2}.

Incorporation of 0.5% $NaNO_2$ brings down C_{dl} values to the maximum extent for (OPC + PSC) followed by (OPC + PPC) and (PPC + PSC) systems in increasing order. So steel in the binary system (OPC + PSC) performs better than in the other two systems. Figure 7 shows the impedance behaviour of steel in ternary cement extract. Table 4 gives values of charge transfer resistance (R_{ct}), double layer capacitance (C_{dl}), corrosion rate and efficiency for steel in ternary system. Steel in the ternary system shows higher R_{ct} values and lower C_{dl} values than all binary systems. Incorporation of chloride ions reduces R_{ct} values to different extents depending on the chloride ion concentration. The addition of 0.5% $NaNO_2$ to the binary system in the absence of chloride ions enhances R_{ct} to a very great extent. But in the presence of different percentages of chloride ions R_{ct} values are reduced to different extents depending on the concentration of chloride ions.

Table 3 Potentiodynamic polarisation parameters for steel in ternary system

System	E_{corr} (mV vs. SCE)	Tafel slopes (mV dec^{-1})		Corrosion rate (mmpy)	Efficiency (%)
		b_a	b_c		
Plain	−544	53	84	0.0010	–
Plain + 1% Cl^-	−566	58	88	0.0042	–
Plain + 2% Cl^-	−576	50	87	0.0062	–
Plain + 3% Cl^-	−580	58	85	0.0091	–
Plain + 0.5% NO_2	−538	50	87	0.0001	92
Plain + 0.5% NO_2 + 1% Cl^-	−547	53	87	0.0004	91
Plain + 0.5% NO_2 + 2% Cl^-	−556	51	85	0.0007	89
Plain + 0.5% NO_2 + 3% Cl^-	−566	51	81	0.0017	81

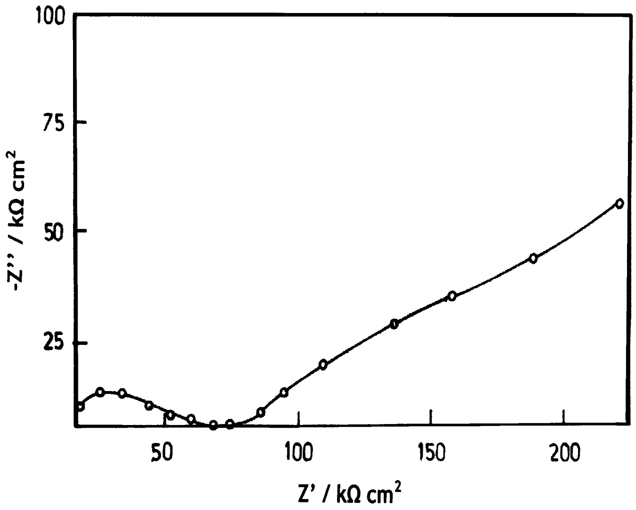

Fig. 7 Impedance behaviour of steel in ternary cement extract

Values of C_{dl} are found to increase with increase in NaCl concentration. But in the presence of 0.5% $NaNO_2$, C_{dl} values decrease to different extents depending on the concentration of NaCl. Corrosion rate of steel in the ternary system are found to increase with increase in chloride. Steel shows a very low value of corrosion rate in the presence of 0.5% $NaNO_2$. But incorporation of different concentration of NaCl increases the corrosion rate. The ternary system containing 0.5% $NaNO_2$ gives 91% inhibition efficiency which decreases in the presence of NaCl of different concentrations. Inhibition efficiency for different systems in the presence of 0.5% $NaNO_2$ and 1% NaCl follows the order:

$$\begin{array}{r}(\mathrm{OPC}+\mathrm{PPC}+\mathrm{PSC})\\ 90\%\end{array} > \begin{array}{r}(\mathrm{OPC}+\mathrm{PSC})\\ 86\%\end{array}$$
$$> \begin{array}{r}(\mathrm{OPC}+\mathrm{PPC})\\ 74\%\end{array} > \begin{array}{r}(\mathrm{PPC}+\mathrm{PSC})\\ 66\%\end{array}.$$

A similar trend has already been observed in weight loss measurements and potentiodynamic polarisation studies.

3.7 Visual observation

Steel dipped in different systems was visually examined for the formation of any rust. Steel in the binary system (PPC + PSC) showed the formation of rust even within a month of exposure. Steel in the (OPC + PPC) system showed the formation of rust quickly after 1 month. But in the (OPC + PSC) system it exhibited rust after 3 months. But steel in the ternary system showed no rust formation even at the end of 6 months.

3.8 Surface coverage versus concentration of chloride

Surface coverage (θ) for different concentrations of sodium nitrite on steel in binary and ternary systems was evaluated from potentiodynamic and impedance measurements as described earlier [19]. Plots of θ versus $\log[Cl^-]$ were obtained and are shown in Fig. 8a, b. Surface coverage decreases with increase in concentration of chloride ions in all cases. Coverage values are found to be maximum for the ternary system ($\theta > 0.9$) even in the presence of higher concentrations of chloride ions indicating the better performance of steel in the ternary system. The greater the surface coverage, the higher the efficiency.

4 Conclusions

The following conclusions can be drawn from the present study.

- The efficiency of different nitrites in reducing the corrosion of steel in binary and ternary cement environments follows the order:

$$NaNO_2 > KNO_2 > Ca(NO_2)_2.$$

- The performance of steel in different cement systems follows the order:

Table 4 Impedance parameters for steel in ternary system

System	R_{ct} (Ω cm^2 × 10^3)	C_{dl} (F cm^{-2} × 10^{-5})	Corrosion rate (mmpy)	Efficiency (%)
Plain	5.103	6.102	0.0019	–
Plain + 1% Cl^-	3.821	6.597	0.0052	–
Plain + 2% Cl^-	3.074	8.332	0.0071	–
Plain + 3% Cl^-	2.666	12.990	0.0092	–
Plain + 0.5% NO_2	12.420	3.841	0.0002	91
Plain + 0.5% NO_2 + 1% Cl^-	6.033	4.234	0.0005	90
Plain + 0.5% NO_2 + 2% Cl^-	5.700	5.284	0.0009	87
Plain + 0.5% NO_2 + 3% Cl^-	4.597	5.847	0.0017	82

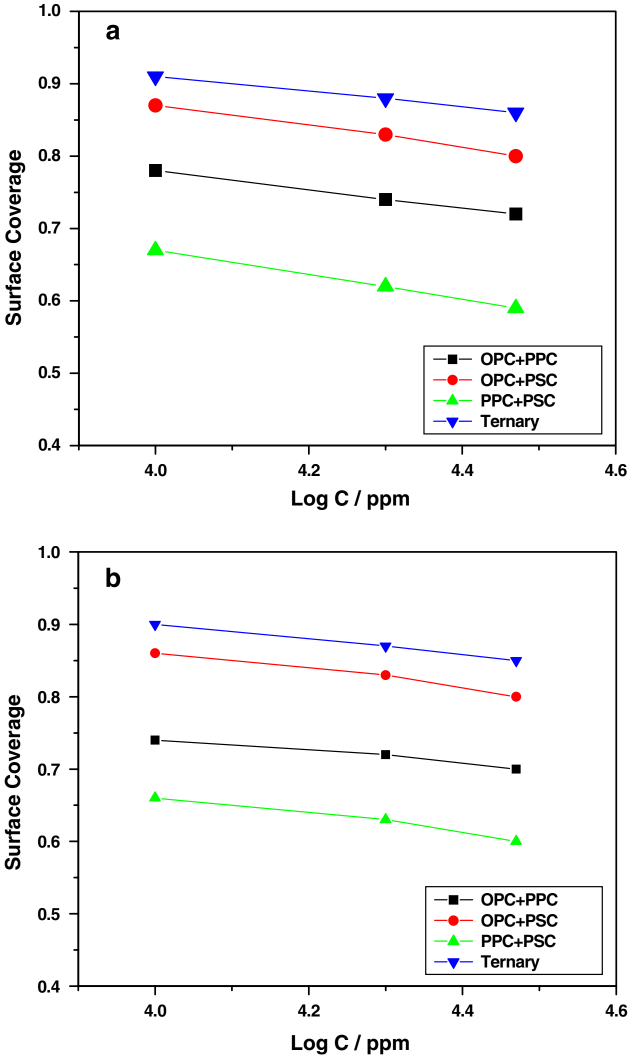

Fig. 8 Relationship between concentrations of chloride and surface coverage values from (**a**) polarisation experiments and (**b**) impedance experiments

Ternary > (OPC + PSC) > (OPC + PPC) > (PPC + PSC).

- Maintenance of passivity of steel in different systems for a duration of 6 months follows a similar order as above.
- The use of binary and ternary systems does not affect the alkalinity of cement.
- Values of corrosion rate and inhibition efficiency obtained from conventional weight loss measurements and different electrochemical techniques are found to be comparable.
- All the studies bring out the fact that composite cement, especially the ternary system, provides better protection for steel in concrete.
- As all the varieties of cement, namely OPC, PPC and PSC are commercially available, it is practically possible to prepare composite binary, ternary cement systems in both laboratory and field conditions.

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