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Influence of Surfactants on Electrochemical Chloride Extraction in Reinforced Cement Mortar

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This paper aims to examine the effect of surfactants on the electrochemical chloride extraction for remediating the chloride contaminated reinforced cement mortar. Triethanolamine dodecyl sulfate (TADS) and sodium dodecyl benzene sulfonate (SDBS) were chosen to represent as cationic surfactant and anionic surfactant, respectively. Different applied potentials of 20V, 40 V and 60V were applied. The extracted chlorides in electrolytes, residual chlorides in mortar, energy consumption, corrosion potential and corrosion current density (decided by half-cell potential and potentiodynamic polarization) of embedded steel were measured. The results indicate that the surfactants increase obviously the efficiency of electrochemical chloride extraction. Compared to SDBS, TADS has a better effect of chloride removal. Besides, the surfactants reduce the energy consumption. Furthermore, the decreases of the I_{corr} values and increases of the E_{corr} values for the embedded steels due to the additions of surfactants are obtained after the electrochemical treatments. Accordingly, the surfactants can be applied to improve the remediation effect of electrochemical chloride extraction on the chloride-contaminated concrete structures in real environment.

Keywords: Electrochemical chloride extraction; chloride extraction efficiency; surfactants; corrosion potential; corrosion current density.

1. INTRODUCTION

Chloride-induced corrosion of steel is one of the main forms of damage to reinforced concrete structures exposed to chloride environments, which leads to a decrease in structural strength and service life, resulting in huge economic losses and maintenance costs. There are two ways for chloride-induced corrosion of steel to damage reinforced concrete structures. One is that the corrosion of the reinforcing steel reduces the cross-sectional area of the steel and reduces the structural strength of the reinforced concrete. The other is that the corrosion products generated by steel corrosion accumulates in the concrete pore space, which build up hoop stresses around the rebar, which causes the reinforced concrete to crack and eventually destroy the structure[1].

For addressing this issue, it is a great of practical significance to remove the aggressive chloride ions from the chloride-contaminated reinforced concrete structure for upgrading its durability. Electrochemical chloride extraction (ECE) treatment as a non-destructive technology has been widely used for remediating the chloride-contaminated reinforced concrete[2-4]. The technology applies DC electric field between concrete and steel to generate a potential gradient that drives the chloride ions out of the contaminated reinforced concrete. After the ECE treatment, the chloride content near steel will be remarkably decreased and the further corrosion of steel will be avoided[5].

Despite this, it has to be pointed out that the ECE treatment can not completely remove the aggressive chloride ions from the chloride contaminated concrete structure[6-8]. Only 40–60% of the total chlorides can be removed, as shown in the literatures[9-11]. Although some measures including prolonging the treatment duration and increasing the applied current density can be applied to raise the chloride removal efficiency, some other problems can be induced, and the results are not yet satisfactory[12-16]. The residual chlorides after the ECE treatment will continue to threaten the durability of concrete structures. It is significant for us to seek new ways to increase further the chloride removal efficiency of electrochemical chloride extraction.

Surfactants had been extensively used to enhance the removal efficiency of heavy metals, organic compounds and fluorine from soils during electrokinetic remediation[17-20]. They are introduced to change the direction and magnitude of electroosmosis flow and decrease the Van der Waals interactions between pollutants and soil particles[21, 22]. The ECE treatment in nature belongs to the technology of electrokinetic remediation. In addition, the physical adsorption of chlorides onto the various hydrates of binder is mainly dominated by the Van der Waals interactions. Based on this, it is anticipated that the surfactants may exert an effect on the chloride removal efficiency of electrochemical chloride extraction.

This paper aims to examine the effects of surfactants on the electrochemical chloride extraction for remediating the chloride contaminated reinforced cement mortar. Triethanolamine dodecyl sulfate (TADS) and sodium dodecyl benzene sulfonate (SDBS) were chosen to represent as cationic surfactant and anionic surfactant, respectively. Different applied potentials of 20V, 40 V, 60V were applied. The extracted chlorides in electrolytes, residual chlorides in mortar, energy consumption, corrosion potential and corrosion current density (decided by half-cell potential and potentiodynamic polarization) of steel reinforcement were measured.

2. EXPERIMENTAL

2.1. Materials and sample preparation

Table 1. Oxide composition of cement (% w/w).

Oxide	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	Na_2O	Total Cl	SO_3	Ignition loss
Composition (wt. %)	57.27	24.99	9.32	3.11	0.86	0.83	0.28	0.05	1.13	2.16

In this study, No. 42.5 ordinary Portland cement (OPC) was used. Its oxide composition was indicated in Table 1. River sand with a fineness modulus of 2.3 was used as fine aggregate. Tap water and deionized water were used to prepare mortars and various electrolytic solutions, respectively.

The mortar samples with the size of 4 cm×4 cm×16 cm were fabricated by using the water/cement ratio of 0.5 and sand/cement ratio of 2.5. One plain steel rebar with 1 cm diameter and 10 cm long was embedded in the centre of mortar sample. For simulating the chloride contaminated concrete, sodium chloride (analytical reagent, 3.0 % by mass of cement) was added to the tap water during mixing. Moreover, the mortar samples were demolded after 24 h of casting and then cured in a 95% humidity chamber at 20 ± 2°C for 28 d.

2.2. ECE treatments

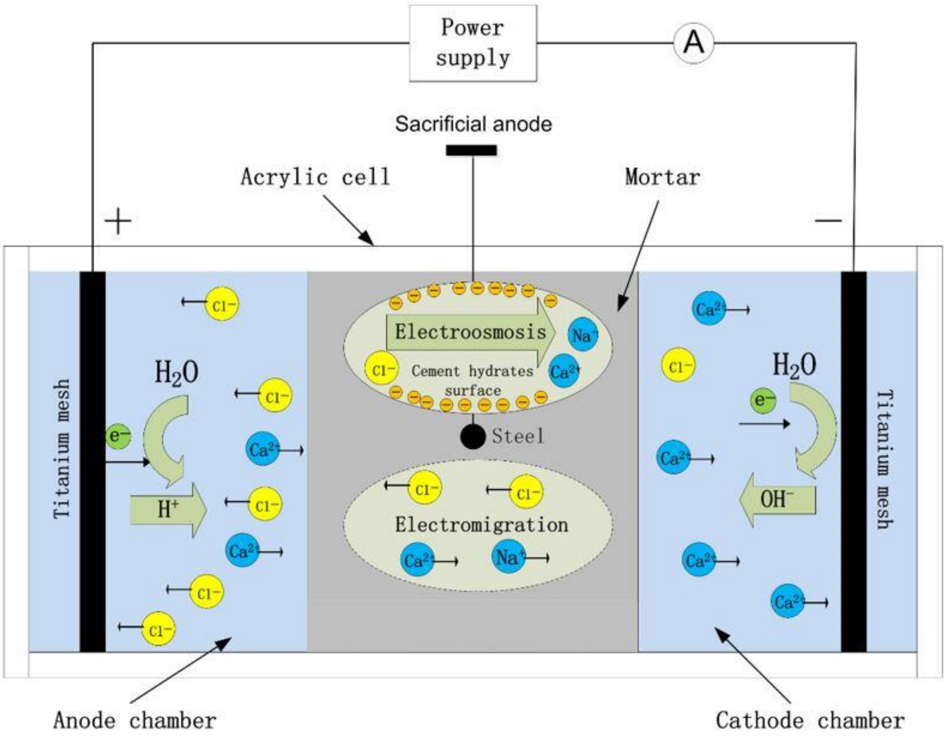

Figure 1. Schematic diagram of the experimental setup.

The schematic of the experimental setup is shown in Fig.1. The mortar sample was installed in the middle of Perspex electrolytic tank (24 cm×15 cm×5 cm). The left and right parts were served as anode chamber and cathode chamber, respectively. Platinized titanium meshes were applied as electrodes and electrically connected to a DC power supply in the ECE treatment. Such a setup was beneficial to evaluate comprehensively the effects of surfactants on the electrochemical chloride extraction, especially the individual contributions due to the electroosmosis and electromigration actions. For protecting the steel against the stray current corrosion during the ECE treatment, the embedded steel was connected to the magnesium sacrificial anode by electric wire. In addition, the sacrificial anode was buried in the ground as the earthing electrode for the stray current[23]. In previous paper, it had been proved that a good effect of steel corrosion protection could be attained[24].

The solutions were identical in the anode chamber and cathode chamber, which had three types. The first type was the saturated calcium hydroxide solution. The second one was the saturated calcium hydroxide solution containing the cationic surfactant with the content of 0.5% by mass of electrolyte, and the third one was the saturated calcium hydroxide solution containing the anionic surfactant with the content of 0.5% by mass of electrolyte. Triethanolamine dodecyl sulfate (abbreviated as TADS, C18H41NO7S, Mr=415.59) and sodium dodecyl benzene sulfonate (abbreviated as SDBS, C18H29NaO3S, Mr = 348.48) were chosen to represent as the cationic surfactant and anionic surfactant, respectively. The applied potentials were 20, 40 and 60 V, respectively. ECE treatments of all mortar samples containing sodium chloride (3.0 % by mass of cement) were operated for 28 days at the ambient temperature.

2.3. Measurements

2.3.1 Extracted chlorides in electrolytes

The anode and cathode electrolytic solutions were collected periodically for determining the extracted chloride amount every two days during the ECE treatments. The extracted chloride contents in the electrolytic solutions were measured by potentiometric titrating with silver nitrate ($AgNO_3$).

Furthermore, the total of extracted chloride amounts in two electrolytic solutions was introduced into the following formula to calculate the extracted chloride percentage:

$$w(\%) = \frac{m}{M} \times 100\% \qquad (1)$$

where w is the extracted chloride percentage, M is the initial chloride ion content (3.0 % by mass of cement) and m is the total of extracted chloride content in the anode and cathode electrolytic solutions.

2.3.2 Residual chlorides in mortars

Mortar samples near the steel bars were taken before and after the ECE treatment. Subsequently, the slices were powdered until passing through the sieve of 0.16 mm. After drying, 10 g powders were added into 100 ml distilled waters and 100 ml nitric acid solutions so as to determine the free Cl^- content and total Cl^- content, respectively. The mixtures were vigorously stirred for a period, and then left to stand for 24 h. All the chloride contents were measured by the potentiometric titration with 0.01M silver nitrate ($AgNO_3$).

2.3.3 Energy consumption

During the ECE treatments, the output electric currents for all samples were monitored by a digital multimeter. The energy consumption of the ECE treatment was calculated as follows:

$$\mathrm{P} = \int UI\,dt \qquad (2)$$

where P is the energy consumption (Wh), U is the applied potential (V), I is the output electric current (A) and t is the time (h).

2.3.4 Corrosion potential and corrosion current density

Half-cell potentials (HCPs) for the mortar samples were conducted according to the ASTM C-876[25] guidelines.

Potentiodynamic polarization was applied to evaluate the corrosion properties of the embedded steels before and after the ECE treatments. The embedded steel, platinum electrode and saturated calomel electrode (SCE) were served as the working electrode, the auxiliary electrode and the reference electrode, respectively. Both the cathodic and anodic polarization curves were recorded potentiodynamically using PARSTAT 2273 (AMETEK Corporation, USA). The measurement was carried out with the sweep rate of 1 mV/min and potential range of +200 to -200 mV vs. SCE. All the experiments were performed at the ambient temperature.

3. RESULTS AND DISCUSSION

3.1 Extracted chlorides in electrolytes

The changes of total chloride amounts in the anode and cathode electrolytic solutions with time for the ECE treatments are presented in Fig.2. The extracted chloride amounts in two electrolytes and extracted chloride percentage after the ECE treatment are shown in Table 2.

Table 2. Extracted chloride amounts in two electrolytes and extracted chloride percentages after the ECE treatments

Potential (V)	Electrolyte solution	Initial chloride amount (mg)	Chloride amounts in electrolytes (mg)			Extracted chloride percentage (%)
			Anolyte	Catholyte	Total	
20	$Ca(OH)_2$	9102.5	3177.1	254.6	3431.7	37.7
	$Ca(OH)_2$+TADS	9102.5	4348.0	89.3	4437.3	48.7
	$Ca(OH)_2$+SDBS	9102.5	2484.2	1147.9	3632.1	39.9
40	$Ca(OH)_2$	9102.5	3875.2	331.2	4206.4	46.2
	$Ca(OH)_2$+TADS	9102.5	5266.3	75.9	5342.2	58.7
	$Ca(OH)_2$+SDBS	9102.5	2961.9	1479.8	4459.7	50.0
60	$Ca(OH)_2$	9102.5	4755.7	407.8	5163.5	56.7
	$Ca(OH)_2$+TADS	9102.5	7088.6	76.6	7165.2	78.7
	$Ca(OH)_2$+SDBS	9102.5	3496.2	1874.7	5370.9	59.0

For all the ECE treatments, the most of extracted chlorides happen in the initial 20 days. The amount of extracted chloride is rapidly increased in the initial, then slowly increased and finally attain a stable value, of which such tendency is independent of the applied potentials and surfactants. However, the stable values of extracted chloride amounts are effected by the applied potentials and surfactants. As shown in Fig.2, the stable values are increased when the applied potentials are increased from 20 V to 60 V. It is mainly attributed to the fact that the electromigration and electroosmosis flows are enhanced with the increase of the applied potential in the ECE treatments[26].

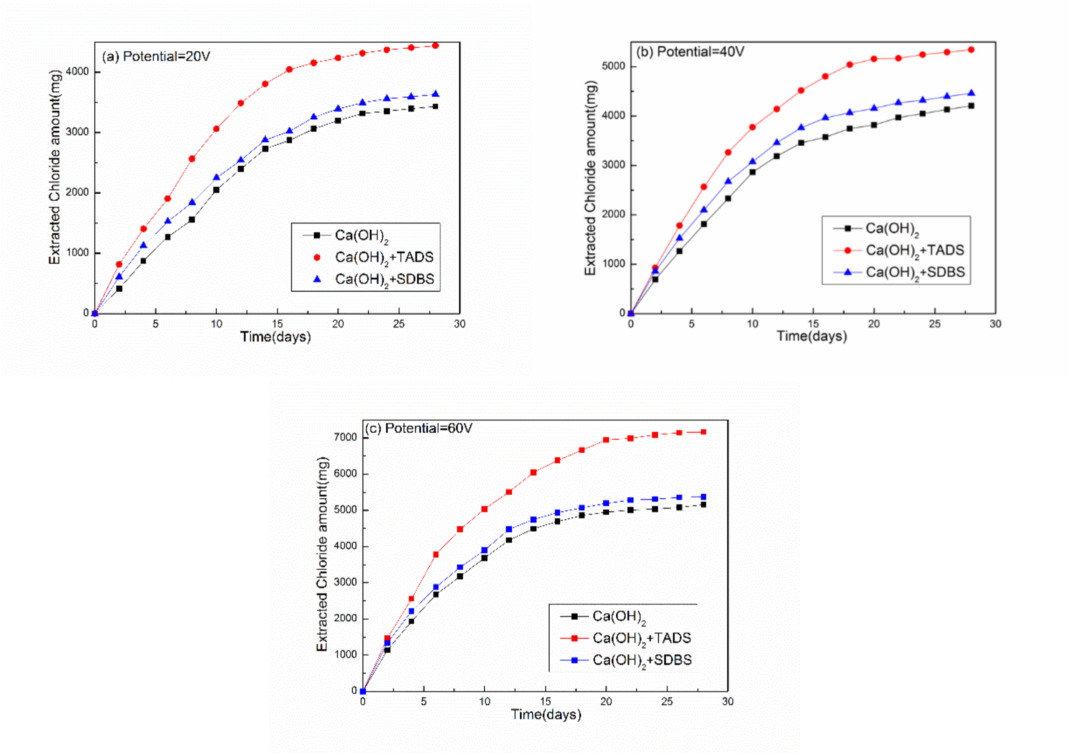

Figure 2. Changes of total chloride amounts in the anode and cathode electrolytic solutions with time, the applied potentials were (a) 20 V, (b) 40V and (c) 60 V, respectively.

In addition, the total of extracted chloride amounts in the anode and cathode electrolytic solutions for the ECE treatments with the surfactants are higher than that for the ECE treatment without surfactant. The cationic surfactant of TADS shows a better performance for chloride removal than the anionic surfactant of SDBS. The extracted chloride percentage for the ECE treatment with the cationic surfactant of TADS even reaches 78.7% when the applied potential is 60 V (see Table 2). In contrast, the percentages for the ECE treatments without surfactant and with the anionic surfactant of SDBS are only 56.7% and 59.0 %, respectively.

Furthermore, the amounts of extracted chlorides in the anode and cathode electrolytic solutions are not identical, as shown in Table 2. In general, the amount of extracted chlorides in the anode electrolytic solution is greater than that in the cathode electrolytic solution. When the anionic surfactant is added, the amount of extracted chlorides in the cathode electrolytic solution is increased. Besides, the amount of extracted chloride in the anode electrolytic solution is reduced. In contrast, for the ECE treatment with the cationic surfactant of TADS, the amount of extracted chlorides in the anode electrolytic solution is increased and that in the cathode electrolytic solution is slightly reduced.

The applied electric field moves the chloride ions in the mortar toward the anode electrode due to the action of electromigration. Also, the action of electroosmosis can move the water flow. The electro-osmotic flow (EOF) is usually transported toward the cathode due to the excess net positive charges in the double layer between the pore solution and mortar pore wall, which is associated with the negative surface charge of cement hydration products[27]. So, the chloride ions in the water flow are carried toward the cathode, and contributed to the chloride content in the cathode electrolytic solution. Kaya and Yukselen[21] have reported that the addition of anionic surfactant increases the absolute value of zeta potential (more negative), which will enhance the EOF. As a consequence, the amount of extracted chlorides in the anode electrolytic solution is increased (shown in Table 2) in the ECE treatment, which leads to the increase of the extracted chloride percentage when the anionic surfactant is used. However, it has to be pointed out that the amount of extracted chlorides in the cathode electrolytic solution is slightly decreased. In contrast, when the cationic surfactant is used, the zeta potential shifts to the positive direction[27]. This change of zeta potential will result in EOF toward the anode. Based on this, the action of EOF moves the chloride ions to the direction same to that by the action of electromigration. This can be proved by the results that the amount of extracted chlorides in the anode electrolytic solution is greatly increased, and that in the cathode electrolytic solution is reduced. Moreover, the cationic surfactant reduces the chloride binding of hydration products and increases the content of free chloride ion in the pore solution[28]. These free chloride ions are easy to be removed under a DC electric field. Thus, once the cationic surfactant is used, the extracted chloride percentage is remarkably increased.

3.2 Residual chlorides in mortars

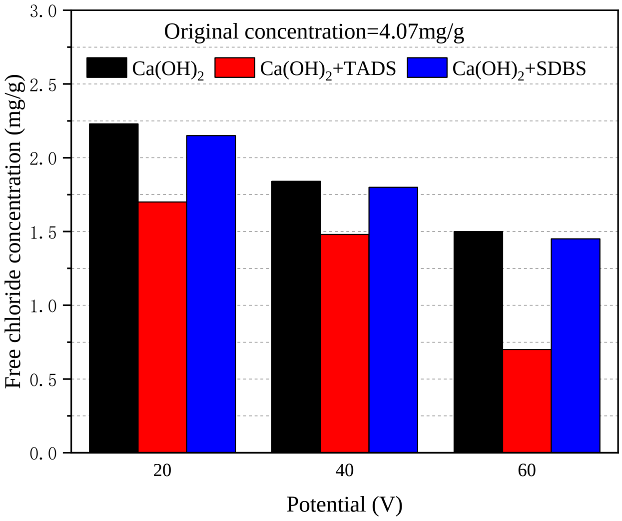

Figure 3. Changes of free chloride concentrations in the mortar sample near the steel bar with the applied potential after ECE treatment.

Fig.3 and Fig.4 show the profiles of free chloride and total chloride in the mortar samples after the ECE treatments, respectively. The residual free and total chloride concentrations are lower than their initial concentrations. After the ECE treatment with the cationic surfactant of TADS, the concentrations of residual chlorides are the lowest, indicating that the efficiency of chloride extraction is the highest.

The efficiency of chloride extraction for the ECE treatment with the anionic surfactant of TADS is slightly higher than that in the ECE treatment without surfactant. For example, after 28 days of the ECE treatments without surfactant at the potential of 40 V, the extracted chloride percentage is 46.2%, but it in the ECE treatment with the cationic surfactant is 58.7%. So, the efficiencies of chloride extraction in all the ECE treatments have the order: cationic surfactant > anionic surfactant > without surfactant.

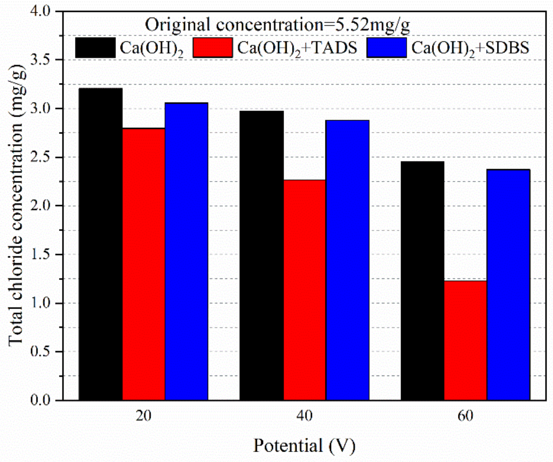

Figure 4. Changes of total chloride concentrations in the mortar sample near the steel bar with the applied potential after ECE treatment.

3.3 Electric energy consumption

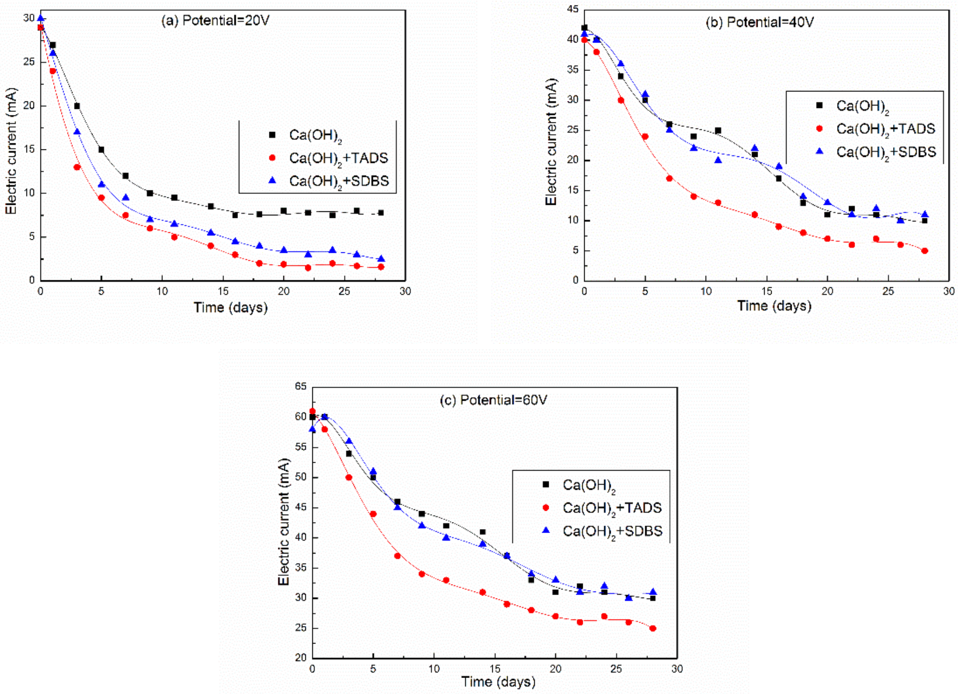

Figure 5. Variations of the electric current with the treatment time, the applied potentials were (a) 20 V, (b) 40V and (c) 60 V, respectively.

The variations of electric current with time during the ECE treatment are shown in Fig.5. As shown, the electric currents exhibit a similar tendency for the ECE treatments with different electrolytic solutions under the same potential, which are initially decreased and finally attain a stable value. The highest electric current at the initial time is due to the largest amount of ions in the pore solution of mortar, especially the dissolution of sodium chloride[29]. As the time goes, the positive or negative ions in the pore solution of mortar move to the external electrolytic solutions by the actions of electromigration and electroosmosis, which results in the increase of mortar resistance[30]. Besides, the electric currents are increased with the increase of applied potential during the initial period of ECE treatment.

The electric energy consumption is an important factor to evaluate the cost-effectiveness of surfactants in the enhancement of electrochemical chloride extraction.

Table 3. Electric energy consumptions for the ECE treatments after treatment at various applied potentials

Potential (V)	Energy consumption (Wh g^{-1})		
	$Ca(OH)_2$	$Ca(OH)_2$+TADS	$Ca(OH)_2$+SDBS
20	44.0	18.4	28.8
40	132.8	71.6	125.2
60	316.4	192.8	302.8

Table 3 shows the electric energy consumptions for the removal of chloride ions from the mortar samples for all the ECE treatments. Higher applied potential can lead to the removal of more chlorides in the mortar samples. However, the energy consumption of per unit chloride ion removal by ECE treatment is also sharply increased with the increase of applied potential. For the control sample, the energy consumption is raised from 11.0 Wh g^{-1} to 79.1 Wh g^{-1} when the applied potential is increased from 20 V to 60 V. Therefore, the increase of applied potential is not one of economical ways to increase the efficiency of ECE treatment. In comparison, the efficiency of ECE treatment is increased, and the power consumption is remarkably reduced when the cationic surfactant is applied. The application of anionic surfactant in the ECE treatment exhibits a similar effect. However, the effect for the application of anionic surfactant is relatively weaker than that for the application of cationic surfactant.

3.4 Corrosion properties of steel after the treatments

3.4.1 Half-cell potential

Fig.6 shows the HCPs of the embedded steels in the mortar samples by the ECE treatments. It can be found that prior to the ECE treatments, the HCP of the steel is -468 mV vs. CSE, indicating that the steel has a high corrosion risk. During the ECE treatments, the values of HCPs for the mortar samples shift to more negative values, which lie in the range of from -700 mV to – 800 mV vs. CSE. Besides, the applied potential has no obvious effect on the value of HCP. Furthermore, the HCPs of embedded steels are obviously increased when the ECE treatments are finished. For example, at the applied potential of 60 V, the HCPs of embedded steels have the values of -198 mV, -131 mV and -163 mV vs. CSE for the samples by the ECE treatments with the electrolytic solutions of $Ca(OH)_2$, $Ca(OH)_2$+TADS and $Ca(OH)_2$+SDBS, respectively. These values of the HCPs are lower than -200 mV vs. CSE, indicating that the passivation of steel is occurring.

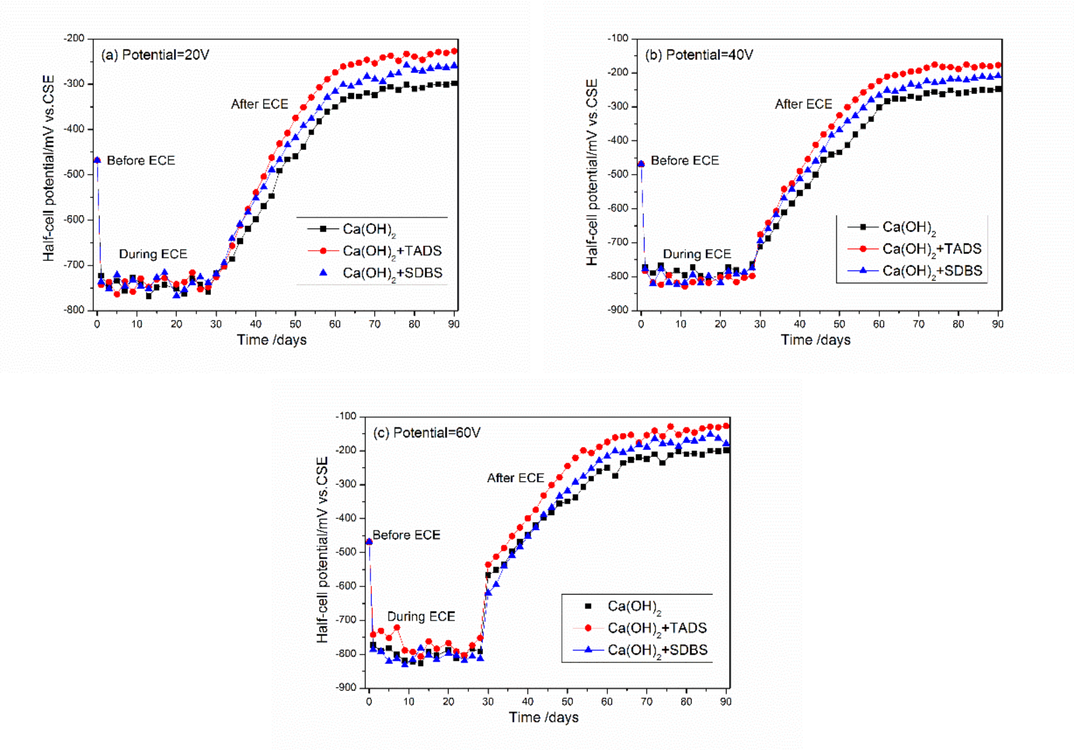

Figure 6. HCPs of the steels in the mortar samples at the applied potentials of (a) 20 V, (b) 40 V and (c) 60 V.

When the applied potential is increased, the HCPs of the embedded steels after the ECE treatments are raised. The reason is due to the fact that the increase of chloride removal amount enhances the stability of the re-passive film on the surface of the steel[10]. Besides, the potentials of steel after the ECE treatments with the surfactants are higher than that after the ECE treatment with the electrolytic solution of $Ca(OH)_2$. Therefore, the surfactants have positive effects on the re-passivation of embedded steel in the mortar samples.

3.4.2 Potentiodynamic polarization

The potentiodynamic polarization curve for embedded steel in the mortar sample before the ECE treatment is given in Fig.7. It is concluded by the traditional extrapolation technique that the corrosion potential (E_{corr}) and corrosion current density (I_{corr}) of embedded steel are -498 mV vs. SCE and 1.1305×10^{-2} mA/cm^2, respectively. The I_{corr} value is far higher than the critical value of 0.1µA/cm^2, which is often applied to identify the corrosion initiation of embedded steel in concrete. Accordingly, it can be decided that the active corrosion of embedded steel is occurring.

Fig.8 a-c show the potentiodynamic polarization curves of embedded steels in the mortar samples after the ECE treatments with various electrolyte solutions (Ca(OH)$_2$, Ca(OH)$_2$+TADS and Ca(OH)$_2$+SDBS) and potentials (20 V, 40 V and 60 V). The corresponding corrosion properties of embedded steels are given in Table 4.

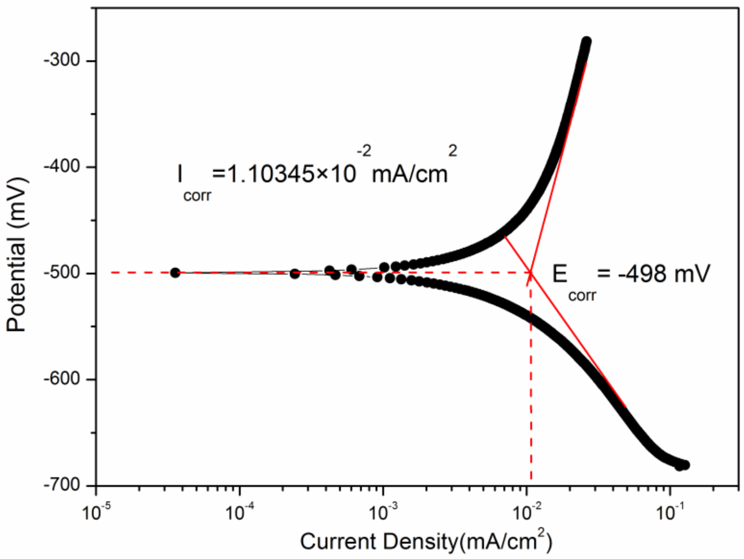

Figure 7. Potentiodynamic polarization curve for embedded steel in the mortar sample before the ECE treatment.

Table 4. Corrosion properties of the embedded steels after the ECE treatments with various electrolytic solutions and applied potentials

Potential	Corrosion properties	Electrolytic solutions		
		Ca(OH)2	Ca(OH)2+TADS	Ca(OH)2+SDBS
20V	Ecorr (mV)	-337	-277	-311
	Icorr mA/cm2×10-3	1.2082	0.7466	0.8281
40V	Ecorr (mV)	-232	-189	-217
	Icorr mA/cm2×10-3	0.9283	0.5698	0.6782
60V	Ecorr (mV)	-116	-86	-101
	Icorr mA/cm2×10-3	0.6179	0.4087	0.4704

After the ECE treatment with the electrolytic solution of saturated Ca(OH)$_2$, the I_{corr} values for the samples are 1.2082×10^{-3} mA/cm^2, 0.9283×10^{-3} mA/cm^2 and 0.6179×10^{-3} mA/cm^2 at the applied potentials of 20V, 40V and 60 V, respectively. Compared to the I_{corr} value before the ECE treatment, the I_{corr} value after the ECE treatment is significantly decreased. In addition, the values of E_{corr} are -337 mV, -232 mV and -116 mV at the applied potentials of 20 V, 40 V and 60 V, respectively. The values of E_{corr} after the ECE treatment are increased by the values of +161 mV, +266 mV and +382 mV vs. SCE relative to that before the ECE treatment. Based on this, it is decided that the corrosion risks of the embedded steels are effectively reduced after the ECE treatment. During the ECE treatment, OH$^-$ ions gather around the steel surface by the action of electromigration, and oxygen is transported into the inside of mortar sample, which lead to the re-passivation of embedded steel[31](Ribeiro, 2013). Thus, the I_{corr} of steel is obviously decreased and the E_{corr} of steel is increased.

Furthermore, it is found that the I_{corr} values of the embedded steels after the ECE treatments with surfactants are lower than that after the treatment with the electrolytic solution of saturated $Ca(OH)_2$ at the identical applied potential. For example, the I_{corr} values of the embedded steels at the applied potential of 40 V are 0.5698×10^{-3} mA/cm^2 and 0.6782×10^{-3} mA/cm^2 for the samples by the ECE treatments with TADS and SDBS, respectively. The values are lower than that for the ECE treatment with the electrolytic solution of saturated $Ca(OH)_2$. Besides, the E_{corr} values of the embedded steels after the ECE treatments with TADS and SDBS are -189 mV and -217 mV vs. SCE, respectively. These values are higher than that after the treatment with the electrolytic solution of saturated $Ca(OH)_2$.

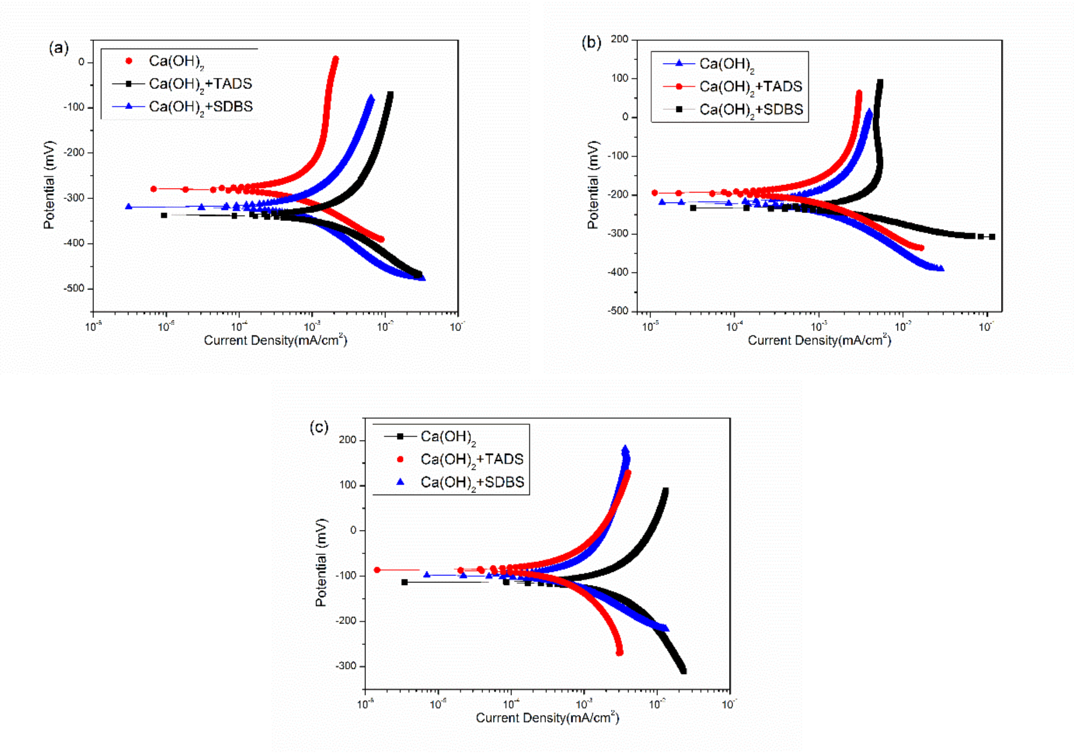

Figure 8. Potentiodynamic polarization curves for embedded steels in the mortar samples after the ECE treatments with various electrolyte solutions at the different potentials of (a) 20 V, (b) 40 V, and (c) 60 V.

From the results, the ECE treatments with the surfactants exhibit better effects on the rehabilitation of chloride contaminated reinforced concrete relative to the ECE treatment with the electrolytic solution of saturated $Ca(OH)_2$. Especially, the application of TADS in the ECE treatment is strongly suggested due to the better effect relative to the application of SDBS.

4. CONCLUSION

An enhanced electrochemical chloride extraction method to repair the chloride-contaminated reinforced cement mortar by surfactants is studied. Triethanolamine dodecyl sulfate (TADS) and sodium dodecyl benzene sulfonate (SDBS) were chosen to represent as cationic surfactant and anionic surfactant, respectively. Some conclusions can be obtained:

(1) The ECE treatment can be enhanced by the applications of surfactants. The efficiencies of chloride extraction in the ECE treatments have the order: cationic surfactant > anionic surfactant > without surfactant. After 28 days of the ECE treatments without surfactant at the potential of 40 V, the extracted chloride percentage is 46.2%, but it in the ECE treatment with the cationic surfactant is 58.7% . The highest extracted chloride percentage reaches 78.7% in the ECE treatment with the cationic surfactant at the applied potential of 60 V.

(2) The amount of extracted chloride is raised with the increase of applied potential, but it is not one of economical ways due to the increase of energy consumption for the traditional ECE treatment with the electrolytic solution of saturated $Ca(OH)_2$. In contrast, the applications of surfactants in the ECE treatments can not only increase the efficiency of extracted chloride, but also reduce the energy consumption. In addition, the cationic surfactant of TADS exhibits a better effect than the anionic surfactant of SDBS.

(3) The I_{corr} values of the embedded steels after the ECE treatments with the surfactants are lower than that without surfactants. Compared to the ECE treatment without surfactant, the E_{corr} values of embedded steels in the mortar samples after the ECE treatments with the surfactants are shifted to the positive direction. Accordingly, the ECE treatments with the surfactants exhibit better effects on the rehabilitation of chloride contaminated reinforced concrete relative to the ECE treatment without surfactant. Especially, the application of TADS in the ECE treatment is strongly suggested due to the better effect relative to the application of SDBS.

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