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Chloride threshold values to depassivate reinforcing bars embedded in a standardized OPC mortar

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Abstract

The chloride threshold to develop active corrosion of the reinforcing steel does not seem to be a unique value and it depends on several factors, such as concrete mix proportions, cement type, C_3A content of cement, blended materials, water/cement ratio, temperature, relative humidity, steel surface conditions and source of chloride penetration among others. Numerous studies have been already devoted to the study of the chloride threshold value for depassivation of the steel embedded in concrete. One of the reasons found for the scatter is the large number of variables that influence the chloride amount for depassivation. The other reason is the lack of accordance for the definition of the chloride threshold itself, either on the determining parameters (visual observation, corrosion potential or corrosion current) or on the expression of the threshold (as Cl^-/OH^- ratio or by weight of cement or concrete). The present paper presents chloride thresholds that were studied in mortar based on corrosion current measurements and expressed as total, free and Cl^-/OH^- ratio. For this study, mainly smoothed bars were used, but also some ribbed bars were tested. Chloride thresholds in the range of 1.24–3.08% and 0.39–1.16%, by weight of cement, for total and free chlorides, respectively, and in the range of 1.17–3.98 for Cl^-/OH^- ratio were found for chlorides admixed in the mixing water. Active corrosion is considered when, in a small exposed area, the corrosion rate of the rebar is higher than 0.1 $\mu A/cm^2$. The threshold in the case of Cl^-/OH^- results a bit higher than that found in a previous work for synthetic pore solution, although the two types of data can be fitted together finding a good correlation. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Chloride; Threshold; Corrosion; Reinforcement; Corrosion rate

1. Introduction

Reinforcing steel bars embedded in concrete depassivate when a certain amount of chlorides build up in their surrounding. The Cl^-/OH^- ratio seems to be the most accurate parameter to take into account when testing the corrosion onset in reinforced concrete [1]. Hausmann [2] and Gouda [3] were the first in identifying the mean value of Cl^-/OH^- ratio, which is around 0.6 in solutions simulating the concrete pore solution. However, due to the difficulty in measuring OH^- concentration in concrete, the free and total chloride contents by weight of cement or concrete are the other parameters that have been widely used to indicate the corrosion risk.

Numerous authors [2–30] have reported different chloride thresholds to depassivate the reinforcing steel, as can be seen in Table 1. However, in spite of this extensive research no agreement among the values obtained is found. This lack of accordance is due to the existence of several parameters influencing the process, for instance, concrete mix proportions, moisture content in the concrete, temperature, type of cation, C_3A content of cement, blended materials, which may affect, in different manners, the cement binding ability and therefore, the amount of free chlorides able to depassivate the steel, as well as the pH of pore solution.

Another reason for this lack of accordance could be related to the definition of the threshold itself. That is, how depassivation is identified. Some authors consider that depassivation is produced when a certain shift in the corrosion potential is produced [2,3]. Other authors use the visual inspection and identify depassivation with the appearance of rust spots on the steel surface [2,3,25,30]. Finally, others relate depassivation with a certain level in

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Table 1
Critical chloride levels required to initiate the corrosion of the reinforcing steel. Literature data wc: weight of cement.

			Values or intervals			
Conditions	Reference	Environment	Free Cl (% wc)	Total Cl (% wc)	Cl/OH	Depassivation detection method
Solutions simulating the concrete	Haussmann [2]	solution			0.60	shift in corrosion potential visual inspection
Solutions simulating the concrete	Gouda [3]	solution			0.35	anodic polarization, shift in potentials and visual observation
Steel in alkaline solutions with chlorides	Goni and Andrade [10]	solutions			0.25–0.8	averaged corrosion rate
Mortar suspensions	Gouda and Halaka [20]	OPC		2.42		anodic polarization
		BFSC		1.21		
Cements with high or low alkali content	Pettersson [4,22,29]	mortars			2.5–6.0	corrosion rate
		80% RH		0.6–1.8	1.7–2.6	
		100% RH		0.5–1.7	1.7–2.6	
Brit. OPC and Sp. BFSC (Cl added as admixture)	Andrade and Page [24]	OPC			0.15–0.69	corrosion rate
		BFSC			0.12–0.44	
Three OPC mortar (external chloride)	Hansson and Sorensen [27]	100% RH				increase in current density potentiostatic test
		50% RH		0.6–1.4		
Concrete slabs stored in 10% Cl seawater	Pettersson [4,22,29]	concrete			1.8–2.9	corrosion rate
Concrete exposed to external chloride contamination	Lambert et al. [18]	concrete			3.00	corrosion rate
Concrete with added Cl	Gouda and Halaka [20]	OPC		3.04		anodic polarization
		BFSC		1.01		
No pre-cleaning the bars	Gouda and Halaka [20]	OPC		0.60		anodic polarization
Cl added as admixture	Kayyali and Haque [21]	concrete				assuming a threshold Cl/OH value of 0.6, calculation of free chlorides
Medium strength concrete		MS	1.15			
High strength concrete		HS	0.85			
High strength concrete + supplement		HSS	0.80			
High strength concrete + supplement + fly ash		HSSFA	0.45			
Cement with different C3A content	Hussain et al. [14]	concretes				assuming a threshold Cl/OH value of 0.3
C3A content = 2.43%			0.14	0.35		
C3A content = 7.59%			0.17	0.62		
C3A content = 14.00%			0.22	1.00		
Concrete with admixed Cl and externally exposed to Cl	Schiessl and Breit [23]	OPC		0.5–1		macrocells currents
		BFSC		1–1.5		
		FA		1–1.5		
Concrete prisms at marine exposure	Thomas et al. [25]			0.50		visual observation + mass loss
Reinforced concrete prisms with fly ash at marine exposure	Thomas [11]	concrete				mass loss
Fly ash content = 0				0.70		
Fly ash content = 15%				0.65		
Fly ash content = 30%				0.50		
Fly ash content = 50%				0.20		
Concrete slabs with added Cl to various exposure conditions	Hope and Ip [30]	OPC		0.097–0.19		corrosion rate, AC impedance, visual inspection, gravimetric mass loss

the corrosion current [4,10,18,22–24,30]. Among these, some use the detection of the increase in the galvanic current as the indicator of depassivation [23] and others use the direct measurement of the corrosion rate to indicate the loss of passivity [4,10,18,22,24,30]. In addition, in some cases, the tests carried out to determine the threshold value have been made not in a "free" corrosion potential, but in potentiostatic conditions [27] or by anodic polarization [3,20]. The detection technique used in each case in determining the moment of the depassivation is also given in Table 1 together with the critical chloride level found.

The data of several authors taken from field and laboratory studies in mortars and concrete indicate that total chloride thresholds may vary by more than one order of magnitude (0.15% to 2.5% by weight of cement) as pointed out by Glass and Buenfeld [13] who made a review of data from several authors.

In the present paper, chloride thresholds are identified by means of the measure of the corrosion rate through

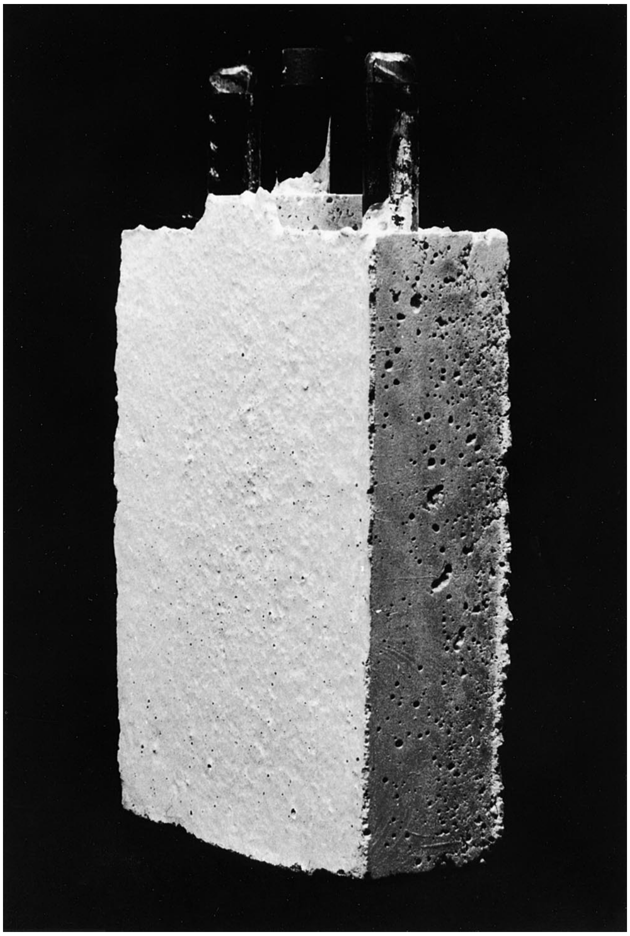

Fig. 1. Aspect of the mortar specimen.

the polarization resistance technique. Depassivation is considered when, in a surface of steel of 1 cm^2, the corrosion intensity is higher than 0.1 μA. This threshold has been selected on the basis of previous works [31–33]. In Ref. [32], it was found that at the bottom of the pit, the pit penetration rate may be up to 10 times higher (1 $\mu A/cm^2$). Smaller values of pit growing rate may result into repassivation.

The variables studied in present work have been the surface finishing of the bars and the type of cation associated to the chloride ion.

2. Experimental

Two series of mortar specimens were cast having dimensions of 8 × 5.5 × 2 cm. The shape of the specimen employed is given in Fig. 1. Each specimen has embedded two identical steel bars. In the first series, eight specimens with smoothed bars were fabricated, and in the second one, four specimens with ribbed bars were fabricated.

Table 2
Portland cement of the type I/45 A, composition in percent (%)

Composition (%)									
R.I	SiO_4	Al_2O_3	Na_2O_3	CaO	MgO	SO_3	P.F.	Na_2O	K_2O
1.92	18.32	5.43	3.28	61.34	1.51	3.08	3.13	0.15	1.04

Table 3
Chemical composition of reinforcement 400 MPa in percent (%)

Composition (%)							
C	Si	Mn	P	S	Cr	Ni	Cu
0.19	0.16	0.78	0.003	0.036	0.16	0.20	0.58

Ordinary Portland cement (OPC), whose analysis is shown in Table 2, was used in all the specimens. A cement/sand ratio of 1/3 and water/cement ratio of 0.5 were used. A standard grading Spanish sand was employed. The steel bars have 8-cm length. The exposed area of the rebar was 3.2 cm^2 for smoothed bars and 5.6 cm^2 for ribbed bars. The smoothed bars were prepared by machining a normal ribbed bar of 6 mm apparent diameter, until the ribs disappear, so that only the steel surface was modified but keeping identical composition of both bars given in Table 3. One end of the bar was embedded in the mortar, while the other side emerged from the mortar specimen to facilitate electrical contact. The embedded end and the steel interface mortar–air were isolated with an isolating tape. A mortar cover of 0.5 cm was used for all the specimens. The mortar specimens were maintained in 100% RH in all test duration.

Chloride ions were added in the mixing water as NaCl and $CaCl_2$. The amount of chloride introduced in each specimen is given in Table 4.

2.1. Electrochemical measurements

A saturated calomel electrode (SCE) was used as reference electrode (RE). The counter electrode consisted

Table 4
NaCl and $CaCl_2$ added to the specimens in the mixing water

Series	NaCl (% by weight of cement)	$CaCl_2$ (% by weight of cement)
Smoothed bars		
1	0.79	–
2	1.18	–
3	1.32	–
4	1.85	–
5	2.37	–
6	2.84	–
7[a]	–	5.38
8	5.14	–
Ribbed bars		
9	0.79	–
10	1.96	–
11	3.49	–
12	5.14	–

[a] Specimen with calcium chloride.

Table 5
Chloride content and Cl^-/OH^- ratio of the specimens with smoothed and ribbed bars

Series	Chloride content (% by weight of cement) and Cl^-/OH^- ratio (pore solution)		
	Free chloride	Total chloride	Cl^-/OH^- ratio
Smoothed bars			
1	0.09	0.48	0.26
2	0.17	0.72	0.39
3	0.23	0.8	0.70
4	0.41	1.12	0.95
5	0.36	1.44	1.03
6	0.67	1.72	1.59
7[a]	0.55	1.72	1.49
8	1.09	3.12	4.74
Ribbed bars			
9	0.13	0.48	0.35
10	0.63	1.19	1.70
11	0.6	2.12	2.14
12	0.97	3.12	4.22

[a] Specimen with calcium chloride.

of an embedded graphite bar. The corrosion potential (E_{corr}), the electrical resistance (R_e) and the polarization resistance (R_p) were recorded periodically, during 90 or 200 days. I_{corr} was calculated through the Stern and Geary equation ($I_{corr} = B/R_p$), where B is a constant. The value given to B in this work was the same employed in Ref. [33], obtained after previous calibration with gravimetric loss. Each I_{corr} value was calculated as the average of the value of the two identical bars embedded in each specimen. A mean value, I_{mean}, was obtained by integrating the I_{corr}–time curve and dividing it by the number of days as was carried out in Ref. [10].

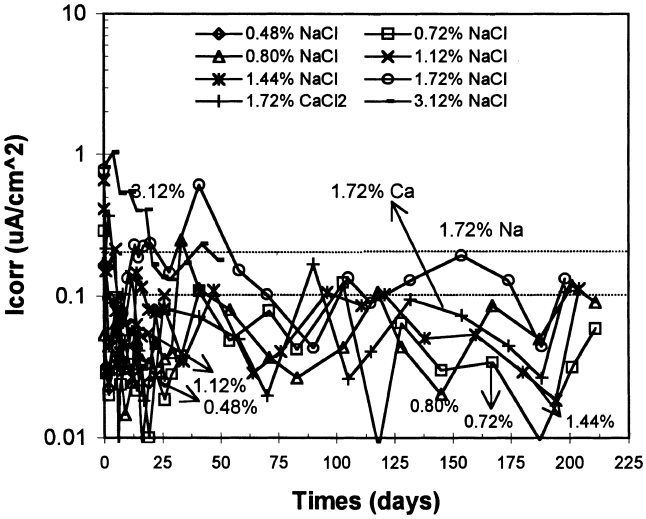

Fig. 2. Influence of total chloride concentration in specimens with smoothed bars. Variation of the instantaneous I_{corr} with time (chloride content is given as total chloride).

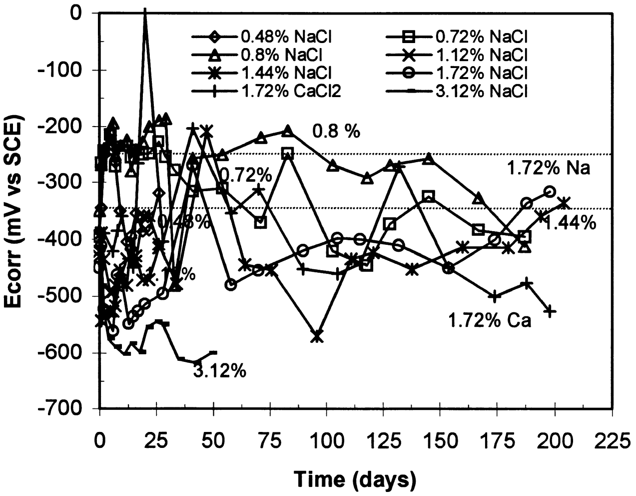

Fig. 3. Influence of total chloride concentration in specimens with smoothed bars. Variation of the instantaneous E_{corr} with time (chloride content is given as total chloride).

2.2. Chloride analysis

The specimens were broken when the I_{corr} do not vary significantly with time. Then, the total chloride content was analyzed by X-ray fluorescence (XRF) and the free chloride and OH^- content by a liquid extraction technique [34]. Three grams of a crushed sample were put in contact with 2 ml of 0.3 M NaOH solution in an inert atmosphere for 24 h. Chloride concentration was then analyzed in the filtrated solution. In the determination of the free chlorides, the establishment of the equilibrium between leaching and the pore solution is assumed. This method has being calibrated before by the pore pressing technique.

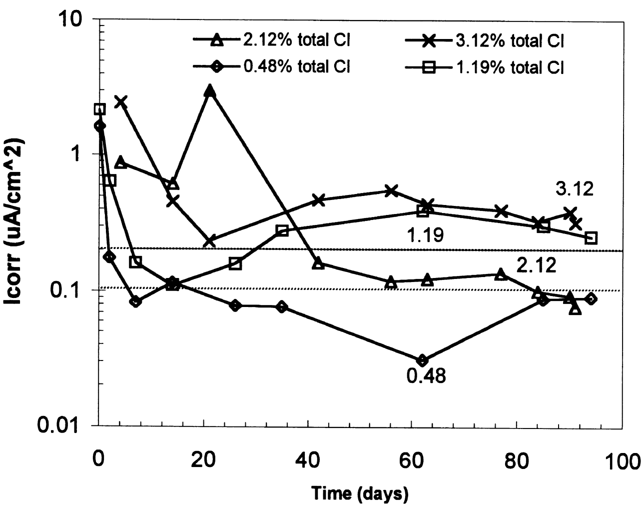

Fig. 4. Influence of total chloride concentration in specimens with ribbed bars. Variation of the instantaneous I_{corr} with time (chloride content is given as total chloride).

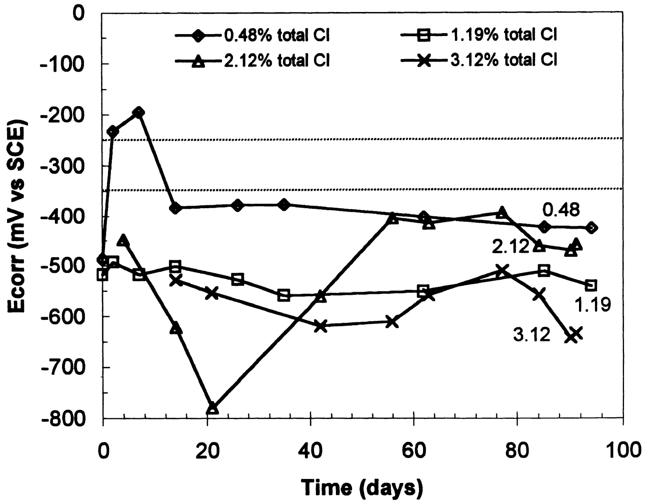

Fig. 5. Influence of total chloride concentration in specimens with ribbed bars. Variation of the instantaneous E_{corr} with time (chloride content is given as total chloride).

2.3. Visual inspection

At the end of the experiments, the bars were removed from the specimen. Then, the presence of rust spots on the steel surface was visually observed. It was noticed that those specimens having I_{corr} values above 0.1 μA/cm^2 showed visible corrosion.

3. Results

Table 5 gives the free and total chloride content values by weight of cement as well as the Cl^-/OH^- ratio in the aqueous phase, which were determined experimentally at the end of the experiment in all the mortars.

Figs. 2 and 3 depict the I_{corr} and E_{corr} values, respectively, along time for the smoothed bars. The 0.1- to 0.2-μA/cm^2 region in the I_{corr} figure separates the regions between active and passive state and the same indicates the lines between −250 and −350 mV in the E_{corr} figure.

Three specimens were broken at early ages to verify the changes observed with the electrochemical measurements (0.48%, 1.12% and 3.12% NaCl). Rust spots were observed in those specimens showing I_{corr} values >0.1 μA/cm^2 for at least some time.

Figs. 4 and 5 present the same data but for the ribbed bars. Now, the I_{corr} values lie in higher levels, with the highest I_{corr} values corresponding to the higher total chloride contents, while those having the smallest chloride content, that is 0.48% of the total chloride, which although active at the beginning, aimed into a passive state. The E_{corr} values of specimens with ribbed bars showed only slightly lower values than those observed for specimens with smoothed bars, with the exception of the specimen containing 2.12% of total sodium chloride added, with ribbed bars, that presents some unexpected cathodic values.

4. Discussion

4.1. Definition of chloride threshold

As already mentioned, the definition of chloride threshold needs some consideration. Depassivation from a practical point of view should mean the developing of an active corrosion that remains with time. This situation cannot be detected by visual observation only because the appearance of colored oxides may take some time to appear or, on the opposite, its appearance may not mean a steady active corrosion. The indication that the corrosion potential (E_{corr}) may give may also be misleading, either because the shift may not be detected or because it may not mean a significant activity.

These uncertainties recommend the use of the indications of the corrosion rate as the most reliable parameter. However, attention has to be paid to the possible relation between corroding and passive areas. If the total exposed area is too large, the ratio of $\sum S_{anodic}/\sum S_{cathodic}$ may be so small that the total corrosion may not mean an increase beyond 0.1 μA/cm^2. In order to detect a significant

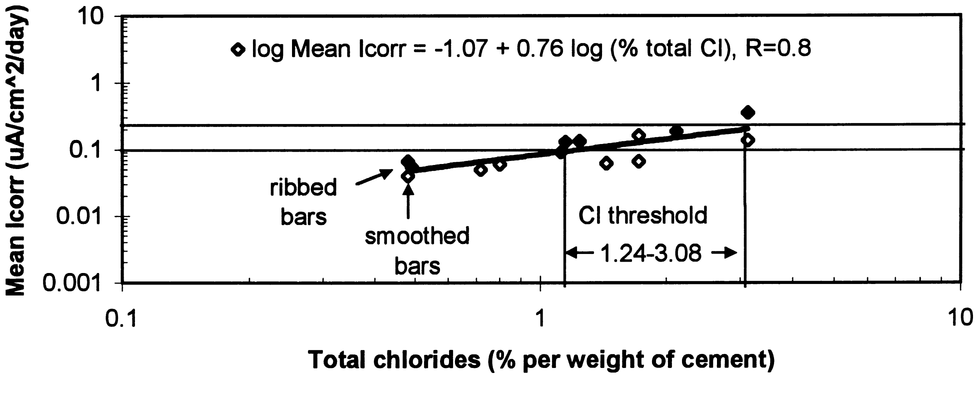

Fig. 6. I_{mean} vs. total chloride. Data correspond to smoothed and ribbed bars together.

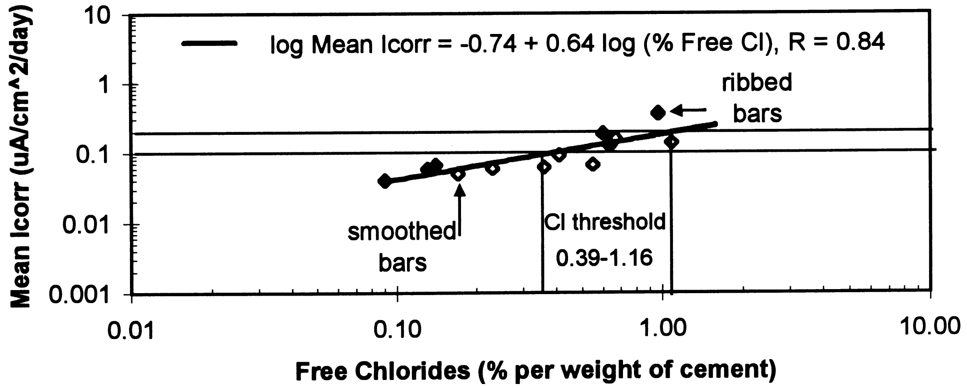

Fig. 7. I_{mean} vs. free chlorides. Data correspond to smoothed and ribbed bars together.

increase, the total area has to be relatively small, as it is the case studied in present paper (around 3 and 6 cm^2 for smoothed and ribbed bars, respectively). With those small exposed areas, assuming that the pit growing rate (penetration depth rate) is around 10 times higher than the measured corrosion rate, an I_{corr} value >0.1 μA/cm^2 means that the value of the pit rate is higher than 1 μA/cm^2, which indicates significant active corrosion not likely to repassivate but become steady.

However, in addition to the above considerations, in the present paper, as made in a previous one [10], the corrosion rate considered is an averaged value during a certain period of time, I_{mean} (μA/cm^2), in order to be sure that the corrosion is steadily active.

4.2. Influence of steel surface and metallurgical conditions on the chloride threshold

Results of Figs. 2–5 showed a bit higher susceptibility of the ribbed bars to be corroded in comparison to the smoothed ones. However, when the I_{mean} is represented vs. chloride (free and total) or Cl^-/OH^- ratio (Figs. 6–8) for both smoothed and ribbed bars, the influence of the steel surface on the chloride threshold results were not significant and they can be fitted together with enough good correlation coefficients.

Although the surface finishing and the metallurgical characteristics (chemical composition and microstructure) seem to influence the rate of corrosion, similar to that stated in Ref. [35], the influence in the chloride threshold level seems less significant, as it is deduced from the results of the paper.

These apparent contradictory results need further research on this aspect.

4.3. Values of chloride threshold

The I_{mean} for the smoothed and ribbed bars together were plotted in Fig. 6 as a function of the total chloride concentration giving a linear trend, as found in Ref. [10].

$$\log I_{mean} = -1.07 + 0.76 \log(\% \text{ total } Cl^-) \quad (r = 0.80)$$

The resulting chloride thresholds are from 1.24% (for 0.1 μA/cm^2) to 3.08% (for 0.2 μA/cm^2) of the total chloride by weight of cement.

These resulting values are of the same order as those obtained by other authors [4,20,22,23,29] under field and laboratory exposure.

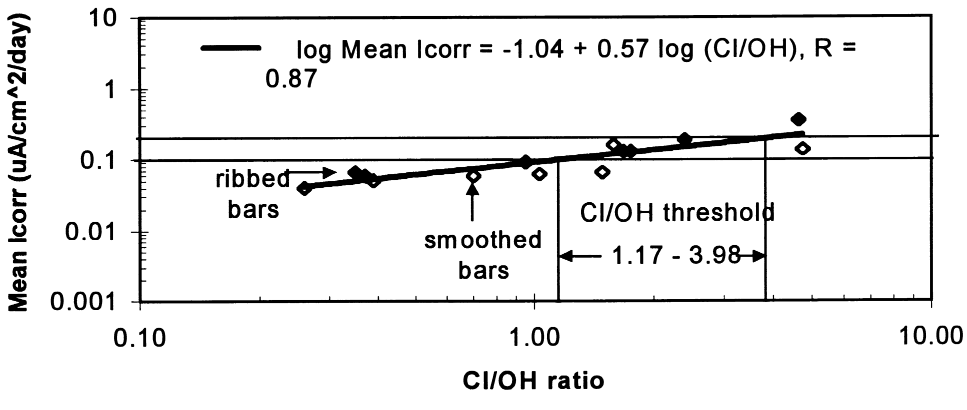

Fig. 8. I_{mean} vs. Cl^-/OH^- ratio. Data correspond to smoothed and ribbed bars together.

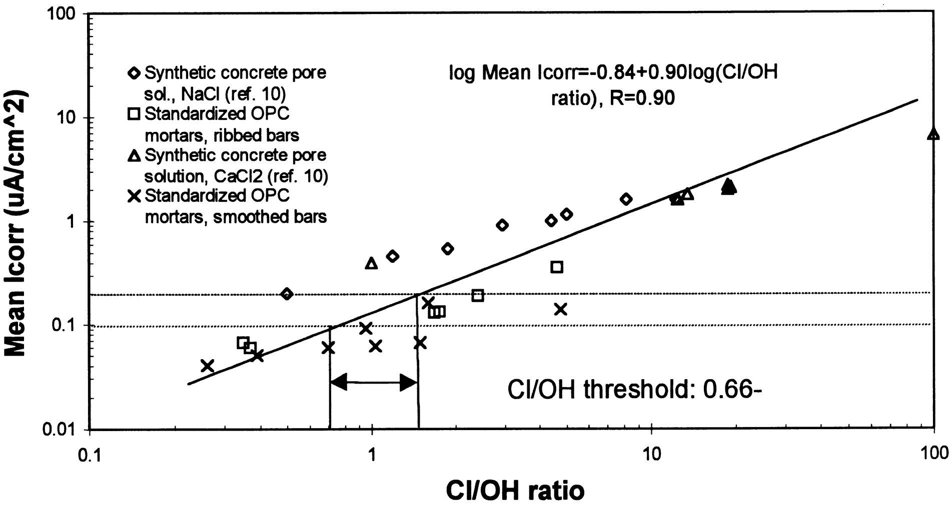

Fig. 9. I_{mean} vs. Cl/OH ratio. Data from Ref. [10] and standardized OPC mortars.

A similar study for determining threshold values have been made in terms of free chloride and $\mathrm{Cl}^-/\mathrm{OH}^-$ ratio. Thus, Fig. 7 shows the relationship found among the mean I_{corr}, as defined above, I_{mean}, and free chlorides in the pore solution (expressed as percentage of free chlorides with respect to the weight of cement). The linear regression obtained is:

$$\log I_{\mathrm{mean}} = -0.74 + 0.64 \log(\%\ \text{free}\ \mathrm{Cl}^-) \quad (r = 0.84).$$

According to these results, the free chloride threshold is in the range from 0.39% to 1.16% of free chlorides. Data from Table 1 show that this threshold of free chloride is also within some of the reported ranges [14,21].

As can be seen in Fig. 8, the same treatment in terms of $\mathrm{Cl}^-/\mathrm{OH}^-$ ratio gives a threshold in the range of 1.17–3.98.:

$$\log I_{\mathrm{mean}} = -1.04 + 0.57 \log(\mathrm{Cl}^-/\mathrm{OH}^- \text{ratio}) \quad (r = 0.87)$$

Data of this value reported by several authors [2–4,10,18,22,24] has been shown in Table 1, with differences of more than one order of magnitude (0.1 to 6) when considering mortar and concrete data. The threshold values reported here are within this range.

It is also important to assess that there is a cation specific effect in the $\mathrm{Cl}^-/\mathrm{OH}^-$ threshold. The series 7 cast with calcium chloride (Table 2) shows a lower $\mathrm{Cl}^-/\mathrm{OH}^-$ ratio (and free chloride) than the specimen 6 with sodium chloride having both the same total chloride content. This has been observed previously by Andrade and Page [24].

Additionally, the results of I_{mean} in the function of the $\mathrm{Cl}^-/\mathrm{OH}^-$ ratio are plotted in Fig. 9 together with the values of Ref. [10] that were obtained in different solutions simulating the pore solution. The linear trend found indicates a threshold value between 0.66 and 1.45.

$$\log I_{\mathrm{mean}} = -0.84 + 0.9 \log(\mathrm{Cl}/\mathrm{OH}) \quad (r = 0.90)$$

Regression obtained in Ref. [10] was: $\log I_{\mathrm{mean}} = -0.43 + 0.62 \log(\mathrm{Cl}/\mathrm{OH})$, being the resulting lower threshold (between 0.25 to 0.8). However, it is also remarkable that the correlation obtained with all the data (solution and

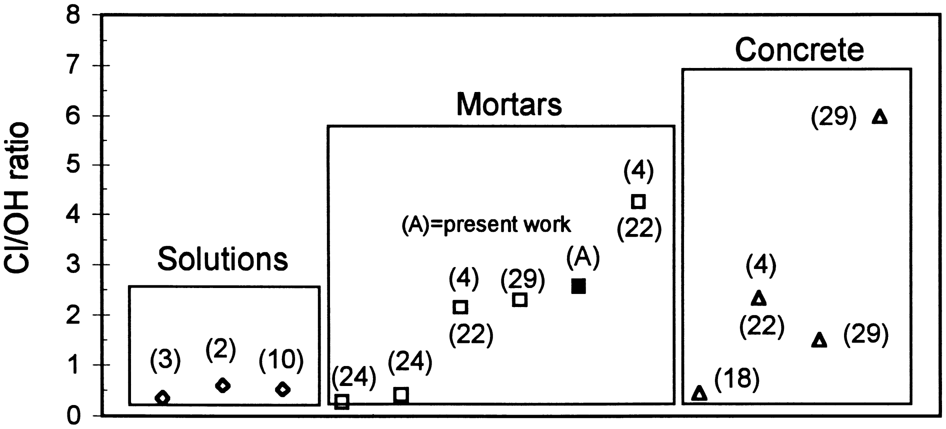

Fig. 10. Mean chloride/hydroxide threshold for solutions, mortars and concretes according to different authors. A: Actual value.

mortar) is quite good, even a bit better ($r = 0.90$) than for mortars only ($r = 0.87$) (Fig. 8).

Finally, some of the data from literature of chloride threshold, given in Table 1, and results obtained in present work, have been plotted in Fig. 10 (as Cl^-/OH^- ratio) to observe the differences existing among the Cl^-/OH^- threshold for solutions, mortars and concretes. In general, the threshold level is higher for mortars and concretes than for solutions. It can be also noticed that the threshold levels between mortars and concretes range in similar orders, from 0.5 to 5.5 for mortar and from 0.5 to 6 for concrete.

5. Conclusions

Based in the definition of chloride threshold as the value that induces permanent active corrosion (mean corrosion currents higher than 0.1 $\mu A/cm^2$), the following conclusions can be deduced from the present study.

1. The ranges of chloride threshold found, expressed in different forms were the following:

% Total Cl^- (weight of cement)	Free Cl^- (weight of cement)	Cl^-/OH^- (pore solution)
1.24–3.08	0.39–1.16	1.17–3.98 0.66–1.45[a]

[a] Solutions and mortars together.

The threshold in mortars, expressed as Cl^-/OH^- ratio, have higher results (1.17–3.98) than those found in synthetic pore solution (0.25–0.8), although the two types of data can be fitted together finding a good correlation that gives a threshold value between 0.66 and 1.45.

2. The linear trends found between I_{mean} ($\mu A/cm^2$) and chloride contents, expressed in different forms were the following:

% Total Cl^- (weight of cement)	Free Cl^- (weight of cement)	Cl^-/OH^- (pore solution)
$\log I_{mean} = -1.07 + 0.76 \log (\% Cl^-)$	$\log I_{mean} = -0.74 + 0.64 \log (\% Cl^-)$	$\log I_{mean} = -1.04 + 0.57 \log (Cl^-/OH^-)$
		$\log I_{mean} = -0.84 + 0.90 \log (Cl^-/OH^-)$

[a] Solutions and mortars together.

3. The type of steel does not seem to influence significantly the threshold value although after depassivation, the I_{mean} in the ribbed bars was slightly higher.

Acknowledgments

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