

## Electrochemical Stability of Steel Reinforced Bars Embedded in Cement Mortars Containing Clinoptilolite as Supplementary

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Received: 20 March 2015 / Accepted: 25 May 2015 / Published: 4 November 2015

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One of the most important characteristic of the Portland Cement (PC) is the alkaline condition that possess, which is situate in passive conditions to the reinforced bar when are embedded in the concrete. The clinoptilolite is a common type of zeolite minerals found in the nature, recently studied in many applications. This paper present a study of behavior of the steel bars embedded in cement mortars containing 0%, 15%, 20%, 25 % of clinoptilolite by weight as replacement of cement. The cement mortars were evaluated in absence and presence of 2% chlorine ions ( $\text{Cl}^-$ ) and were exposed during one year in Relative Humidity (RH)  $\approx$  98% at atmospheric conditions. At this time was studied the corrosion activity employing electrochemical techniques as linear polarization resistance ( $R_p$ ), corrosion current density ( $i_{\text{corr}}$ ), Polarization curves ( $C_p$ ) and the evolution of corrosion potential  $E_{\text{corr}}$ . Furthermore, compressive strength, density and porosity of the mortars were evaluated. The results shows that additions of clinoptilolite until 25% and absence of  $\text{Cl}^-$  not affect the passivity conditions in the steel reinforcement; steel corrosion embedded in presence of  $\text{Cl}^-$  was observed to be similar to the corrosion reported for the mortars reference. Is possible replacement the cement content until 25% by clinoptilolite without affect the mechanical and physical properties in the mortars.

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**Keywords:** zeolite, mortars, corrosion, electrochemical techniques, steel bars.

### 1. INTRODUCTION

The concrete as like other constructions materials is projected with ideas that remain it stable over time, without losing any of its physical and chemical properties that characterize it. Currently

there are a large number of investigations which focus on trying to replace the cement contents for other compounds either natural or artificial, which can reduce costs and the environmental damage caused by the use of cement [1,2].

Previous studies have been focused to find alternative materials to replace the use of cement as binder in construction materials. Natural and synthetic pozzolanic materials as volcanic pumice, volcanic ash, fly ash, rice husk ash, pulverized-fuel ash, silica fume, blast furnace slag have showing good alternatives as binders in these materials [3–8]. Amorphous silica present in these materials has the capacity to react with lime  $[\text{Ca}(\text{OH})_2]$  derived during the hydration processes of the cement forming cementitious materials; this phenomenon gives them their pozzolanic character. The employment of these materials has proven an improvement in mechanical properties, durability and reduction the rate of liberation of heat in the concrete, which is beneficial for mass concrete [9].

One of the natural pozzolanic materials is the clinoptilolite, which is a crystalline phase that belongs to zeolites group. This material is composed by hydrated aluminosilicate of alkali and alkaline cations with microporous framework structure, as the main compound. The structure of clinoptilolite is based on a 3-dimensional skeleton made of silicon tetrahedrons interconnected by oxygen atoms with a part of silicon atoms replaced by aluminum atoms. The amount of silicon oxide in zeolite is usually 70% and may contain around of 12% of aluminum oxide [10]. Thanks to their high specific surface, zeolites are widely used mainly in chemical engineering as catalyst support [11], molecular sieves [12], or sorbents [13]. In civil engineering, their utilization as pozzolans dates back already to ancient times when the mixture of zeolites containing tuff and lime was used as hydraulic binder [14]. In today's building industry, natural zeolites are used mostly as concrete admixtures.

However, provided is very important to study the effect on the physical and chemical properties of the zeolite as material replacement, as well as the damages, which could produce in the concrete and the steel embedded at it in short and long term. One of the main problems in the structures of reinforces concrete is the corrosion of the reinforced steel. Under favorable conditions (free of contaminants), the pH of the concrete keeps in a passivation state or immunity to steel embedded in it. This alkalinity could be affected when a new material is added to replace the cement content. Besides, the diffusion of different ions and in particular chloride ions in cement paste is also an important parameter capable to induce the corrosion of steel bars in reinforced concrete structures.

Different properties in reinforced concrete containing pozzolanic materials have been evaluated and reported such as porosity, density, mechanical properties, chloride diffusion, deterioration by sulfates, corrosion resistance etc. [15-19]. However, despite the number of investigations on the effect of the zeolites in the cement compounds, is important to have more information about the electrochemical behavior of the steel, embedded in these compounds, when are exposed in aggressive and non-aggressive medium at long times.

The aim of this research is study the corrosion behavior of steel rebars embedded in cement mortars, when is added natural pozzolan (clinoptilolite) as partial replacement of the cement with and without the presence of  $\text{Cl}^-$  ions as contaminant. Were tested mortars specimens with different content of clinoptilolite. Electrochemical techniques as lineal polarization resistance, polarization curves and the evolution of corrosion potential were evaluated during one year of exposition at 98% relative

humidity conditions. In addition, porosity, dry density, water absorption and compressive strength were evaluated in order to know the performance of the physical and mechanical properties.

## 2. EXPERIMENTAL

### 2.1 Materials and characterization

An ordinary Portland Cement (PC), zeolite (clinoptilolite), sand and water were used to prepare the mortars. The clinoptilolite used in this study was obtained from the central region of the México. The clinoptilolite was dried at 110 °C for 24 h, crushed and milled until obtain particle between 44 µm and 63 µm. Drinking water supplied of city waterworks was used in this study. The sand used was selected according to the ASTM C778-02.

The reinforcing steel was corrugated bars of 0.95 cm in diameter and 9.0 cm in length with the following chemical composition: 0.47 wt-%C, 0.83 wt-%Mn, 0.18 wt-%Si, 0.021 wt-%P, 0.026 wt-%S, 0.126 wt-%Cr, 0.068 wt-%Ni, 0.040 wt-%Mo, 0.28 wt-%Cu and 0.001 wt-%V. To eliminate the mill scale, the bars were cleaned before embedding in mortar with a 50% hydrochloric acid solution, inhibited with 4 g/L hexamethyltetramine, thoroughly rinsed in running water and then in distilled water and dried immediately with compressed air.

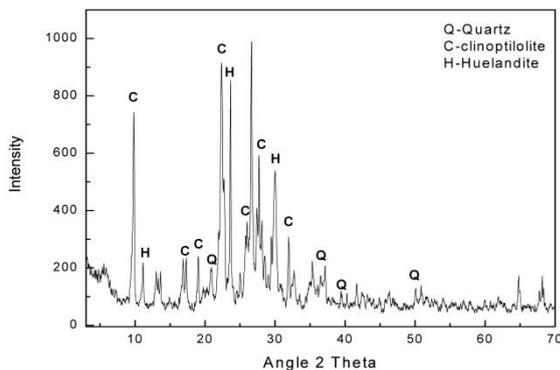
The chemical composition of the zeolite was determined by X-ray fluorescence spectrometer (Bruker AXS). According to the Table 1, the chemical composition of the clinoptilolite showed high contents of silica and aluminum oxides above 90%, followed by potassium and iron mainly. The sum of these oxides exceeds 90% of the total value. Loss of ignition (LOI) was determined according to the method described in ASTM C114-13 [20].

**Table 1.** Chemical composition for the zeolite (clinoptilolite).

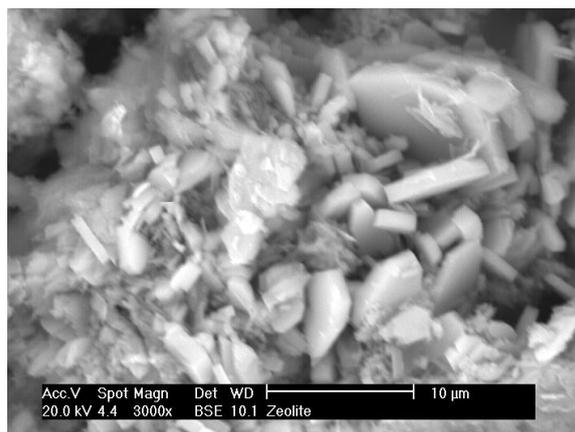
Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	%LOI
ZE	79.89	10.99	1.47	0.72	2.81	0.52	3.29	0.02	9.93

Mineralogical composition of zeolite was determined by X-ray diffraction (XRD) in the 2θ range from 10 to 80°, using a Rigaku DMAX 2200 diffractometer, the X-ray spectrum is showed in Figure 1. The card numbers used to identify the phases present were: 25-1349 of clinoptilolite [(NaKCa)<sub>6</sub>(SiAl<sub>36</sub>)O<sub>72</sub>] as principal crystalline phase, 21-131 of hielandite [CaAl<sub>2</sub>Si<sub>7</sub>O<sub>18</sub>·6H<sub>2</sub>O] as secondary phase and 5-490 of quartz [SiO<sub>2</sub>] in minor content considered as natural impurity in this type of materials [21].

In Figure 2 can be seen the morphologic aspect by SEM (Scanning Electronic Microscopy) corresponding to clinoptilolite structure, can be observed hexagonal plates with sizes between 5-10 µm. The morphological observations were made using a JEOL JSM-6610LV.



**Figure 1.** X- ray diffraction patterns of zeolite showing: Clinoptilolite (C), Huelandite (H) and Quartz (Q) phases.



**Figure 2.** SEM image of morphology structure of clinoptilolite.

## 2.2 Mortars samples

Two types of mortars were prepared with cement/ sand/ water in ratio of 1/3/0.5, the cement content was replacement in 0 %, 15 %, 20 %, 25 % in weight by clinoptilolite. The first type of mortar was used to the mechanicals and physical tests (water absorption, porosity apparent, density), while the second type was prepared to corrosion rate monitoring in the steel rebar. The mortar without zeolite (0 %) was used in all experiment as reference mortar.

Mortar mechanical compressive was determined on 5×5×5 cm cubic specimens prepared; all the mortars were initially cured for 24 hours at 25 °C in a very high humidity atmosphere. After removal from the molds, the mortars were stored in a curing chamber (25 °C, 98% RH) for 7, 14, 28 days [22].

Reinforced mortars were prepared with 0 and 2% chloride ions (sodium chloride was used as a source of Cl<sup>-</sup>) by binder weight to corrosion rate measurements. Prismatic mortar specimens measuring 8×5×2.5 cm, similar to the samples used in prior studies [5,23]; were reinforced with two symmetrically embedded, 0.8 cm round steel bars that worked as test electrodes during measurement

readings, and an 0.5 cm diameter stainless steel wire in a central position that served as the auxiliary electrode. An active area measuring 18.85 cm<sup>2</sup> was delimited on the test electrodes with adhesive tape to insulate the triple steel-mortar-air interface and prevent possible local attacks due to differential aeration [5,23]. Mortars were removed from the molds and cured for 24 hours at 25 °C in a very high humidity atmosphere. After removal from the molds, the mortars were exposed at 25 °C and 98% RH conditions during 365 days.

### 2.3. Measuring techniques

The compressive strength of mortars was measured after 7, 14, 21 and 28 days of curing [24]. For each period of time, three specimens were used in compressive strength measurement for each composition, and the average measurement is the one presented and discussed in this work. Shimadzu Universal Machine UH-X 600 kN was used in all assays.

Water absorption, density and apparent porosity according to ASTM-C-642-06 were determined for mortar samples with 28 days of curing [25].

Corrosion rate assessment was realized employing electrochemical techniques; electrical connection between the counter electrode and the mortar specimens was enhanced by placing a moist sponge between the two during measurement readings. The corrosion potential values were related to the saturated calomel electrode (SCE). All measurements were taken three times.

Three methods were used to monitor steel electrode behavior during 365 day of exposure: frequent determination of the corrosion potential ( $E_{\text{corr}}$ ); lineal polarization resistance ( $R_p$ ) readings to find the corrosion rate pursuant to U.S. standard ASTM C876-09 [26] from which to assess corrosion current densities ( $i_{\text{corr}}$ ) employing the Stern and Geary equation [27] and polarization curves. All the electrochemical measurements were carried out using Gamry reference 3000 potentiostat.

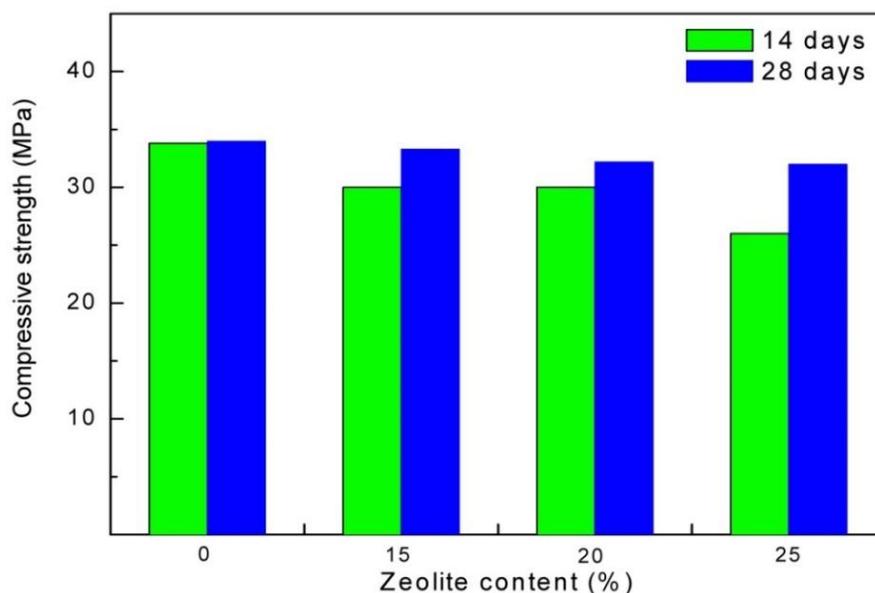
## 3. RESULTS AND DISCUSSION

### 3.1 Compressive strength

In Figure 3 are shown compressive strength mortars results versus the curing time. It can be observed that the reference mortar showed its highest compressive strength values since 14 day of curing, with values of 34 MPa. While the mortars with 15, 20 and 25% of clinoptilolite content showed values of compressive strength of 30, 30 and 26 MPa respectively at 14 days, reaching similar values at the reference mortar (34 MPa) after 28 days of curing. It is clear that regardless of replacement level, clinoptilolite influence in the decrease of mortar strength. At 14 days with 15% and 20% of replacement, this reduction is about 11%, while that 25% replacement is obtained around 23% of reduction.

The partial replacement of cement with clinoptilolite, initially reduces the hardening rate of the mortars, but at later ages the mortars strength with clinoptilolite added, are very similar and sometimes

are higher than the reference mortar. This behaviour could be attributed to the kinetic of the pozzolanic reaction, this reaction is more lower than the cementitious reaction [28].



**Figure 3.** Compressive strength values of the mortars with 0, 15, 20 and 25 % of clinoptilolite in function of curing time.

### 3.2 Physical properties of mortars with clinoptilolite.

The physical properties (water absorption, porosity and dry density) of proposed mortars are presented in Table 2. When the clinoptilolite replace the cement in the mortars, can be see that dry density decreased, showing values from 1512 Kg/m<sup>3</sup> (at the highest clinoptilolite content) to 1678 Kg/m<sup>3</sup> (without clinoptilolite) at 28 days of cured of the mortars. The dry density of the mortars containing clinoptilolite was lower than the reference mortar because of the high porosity and the low specific gravity of clinoptilolite compared to the cement [29]. Respect to the water absorption values, can be noted differences between 2.6% and 12.4% if these values are compared they with the reference mortar. Porosity values of the mortars with clinoptilolite showed an increase from 4.3% to 20.5% after 28 days of curing, compared with the reference mortar. These increases in the values of water absorption and porosity are in relation to the clinoptilolite content in the mortars, can be see that when the clinoptilolite content is increased their physical properties are enhanced too; this behavior is common in the mortars containing zeolites due to the high pores content present in zeolite structure.

Curing time plays an important role in the internal structure of the mortar due to pozzolanic rate reaction; the reaction proceeds as the curing time passes, then, the pores become smaller because of the products formed during the pozzolanic reaction. In the case of the results of the Table 2, the curing time (28 days) is considered short; the pozzolanic reaction is still in process, provoking a high water absorption and porosity in the mortars containing clinoptilolite.

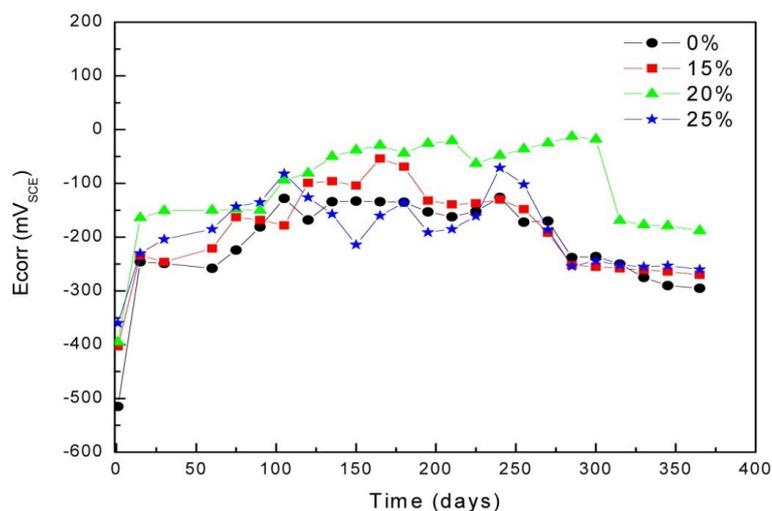
Probably at later ages, the large quantity of reactive SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in clinoptilolite chemically combined with the hydration cement products generate aluminates and CSH gel. These products obstruct the pores presents in zeolite structure enhancing the strength of cementitious materials [30]. However, according to the results in Figure 3 the increase in the porosity of the mortars containing clinoptilolite would not be damaging on their compressive strength, it is expected that as time progresses, the compressive strength values of the mortars would reach values equal or greater than the reference mortar, once the pozzolanic reaction has been carried out completely.

**Table 2.** Physical properties of the mortars with and without clinoptilolite at 28 days of curing.

Clinoptilolite content in mortars (%)	Water absorption (%)	Dry density (g/cm <sup>3</sup> )	Porosity (%)
0	19.3	1678	30.2
15	19.8	1603	31.5
20	21.2	1590	33.1
25	21.7	1512	36.4

### 3.3 Corrosion rate of steel embedded in cement- clinoptilolite mortars

Corrosion potential results were interpreted as specified in standard ASTM C 876-09 [26], corrected for calomel reference electrode which was used.



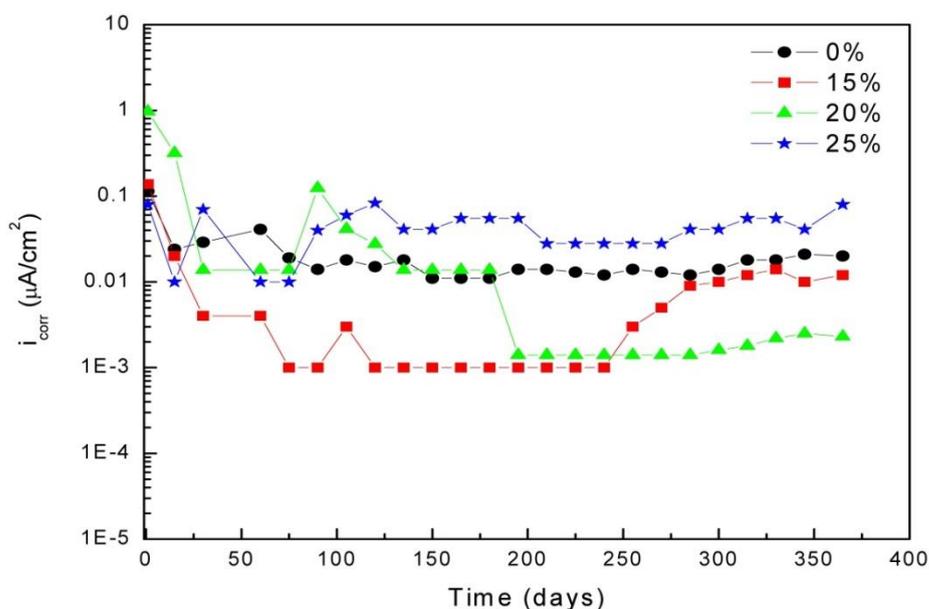
**Figure 4.** E<sub>corr</sub> vs time of exposure of the steel bars embedded in mortar without Cl<sup>-</sup> with different content of clinoptilolite.

Hence, corrosion potential values that were more positive than the threshold -275 mV with respect to saturated calomel electrode (SCE) were interpreted as a passive state of corrosion in the reinforcement area inspected. For other hand, values below than -275 mV depict an active state of the steel corrosion. The determinations of the potential corrosion in the steel-concrete system provide qualitative information of the state of corrosion of reinforcing steel, for this reason is important

complete the information with quantitative determinations which are exposed later in Figures 5, 7, 8 and 9.

Figure 4 show the variation in  $E_{corr}$  values with time of the steel embedded in mortar containing 0%, 15%, 20% and 25% of zeolite without chlorides ions, can be observed that all mortars with clinoptilolite show a similar tendency to each other and the reference mortar, with values over  $-275$  mV indicated a passive state of the reinforcements. These results indicate that the clinoptilolite contained in the mortars not affect the passivity of reinforcement embedded after one year of the exposure in  $HR \approx 98\%$ .

In Figure 5 is possible appreciate the corrosion rate ( $i_{corr}$ ) values, estimated from  $R_p$  measurements taken, respectively, in the reference mortar and the mortars with clinoptilolite after one year of exposure. In this figure can be observe that during the exposure period of time, all mortars show similar  $i_{corr}$  values, lower than  $0.1 \mu A/cm^2$ , maintaining these specimens in the passive state [31].

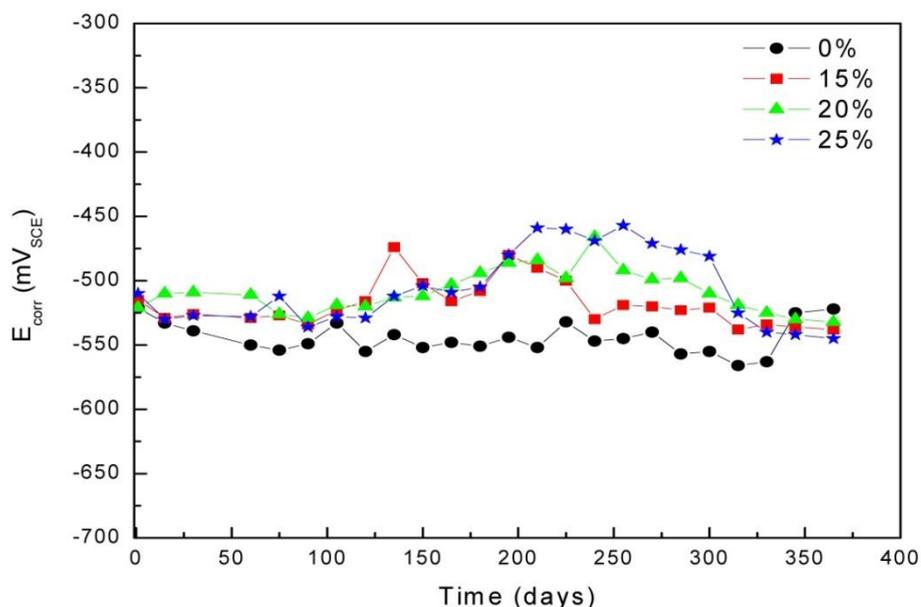


**Figure 5.**  $i_{corr}$  vs time of exposure of the steel bars embedded in mortar without  $Cl^-$  and different content of clinoptilolite.

Figure 6 show the variation in  $E_{corr}$  values with time of the steel embedded in mortar with 0%, 15%, 20% and 25% of zeolite contaminate with chlorides ions. Can be observed that all mortars containing zeolite show a similar tendency to each other and the reference mortar, with values around  $-525$  mV indicates that the corrosion is being carried out over the reinforcements in according to ASTM C 876-09 [26].

In Figure 7 is possible appreciate the corrosion rate ( $i_{corr}$ ) values, estimated from  $R_p$  measurements taken, respectively, in the Portland cement mortar and the mortars containing clinoptilolite after one year the exposure and contaminated with chloride ions. In this figure can be observe that the incorporation of 15%, 20% and 25% clinoptilolite decrease the  $i_{corr}$  values in comparison with the reference mortar. It can be observe that at the beginning of the experiment (first

days of exposure), the mortars containing clinoptilolite showed corrosion rates near to  $10 \mu\text{A}/\text{cm}^2$ , these values are very similar to reference mortar. At this time, the corrosion rate values of these mortars were high due to the passivation process, which implies the formation of a thin film of iron oxides [32]. At 75 days of exposure, all mortars showed corrosion rates close to  $4 \mu\text{A}/\text{cm}^2$ .

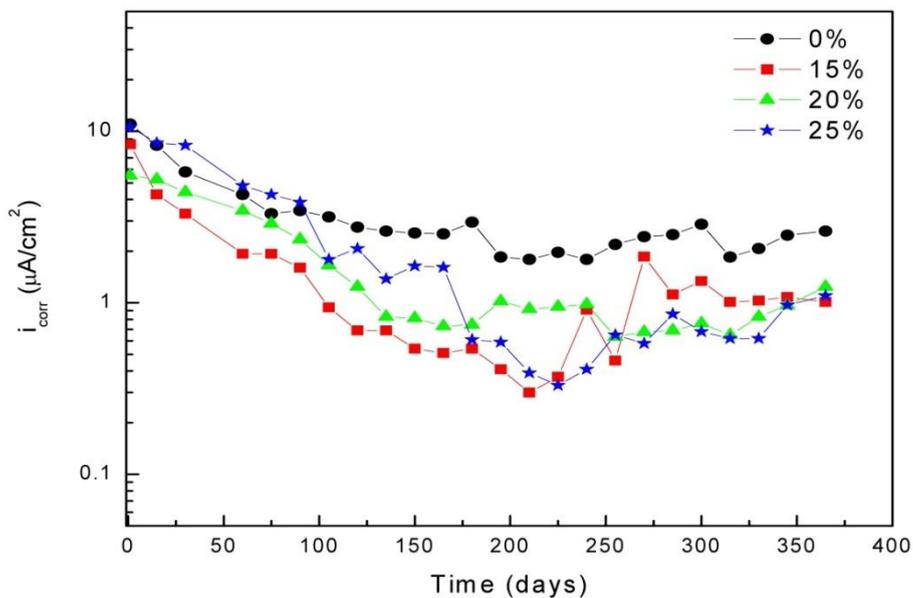


**Figure 6.**  $E_{\text{corr}}$  vs time of exposure of the steel bars embedded in mortar with 2 %  $\text{Cl}^-$  with different content of clinoptilolite.

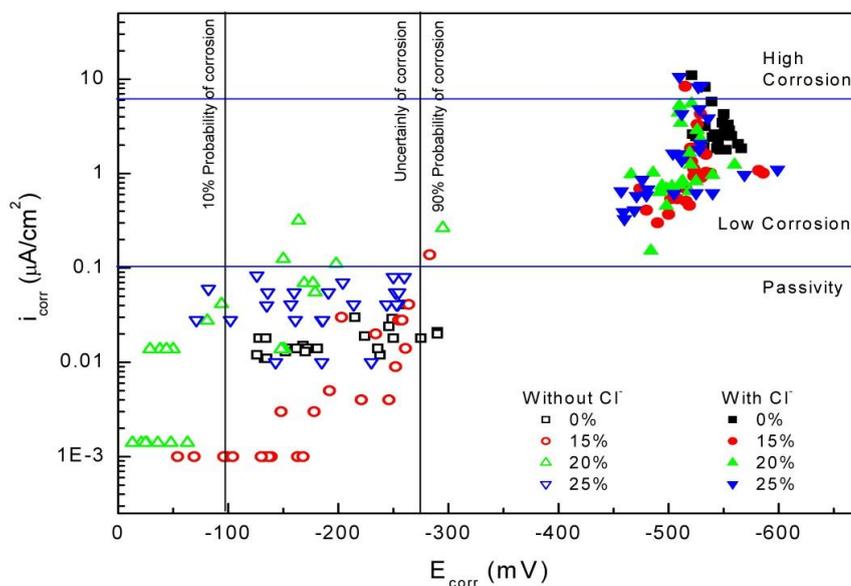
However, after 75 days, the corrosion rate of mortars containing clinoptilolite decreased significantly from  $4 \mu\text{A}/\text{cm}^2$  to  $0.8 \mu\text{A}/\text{cm}^2$ ; at this time the slow development of the pozzolanic reaction is still in progress, modifying the microstructure mortar and consequently, favoring a reduction in the corrosion rate due to enclosure of the pores which avoid the  $\text{Cl}^-$  ions ingress. For other hand, the reference mortar remains almost constant in  $4 \mu\text{A}/\text{cm}^2$  for 365 days of testing indicating a corrosion rate higher than studies mortars. According to the results obtained in this research, it seems that the clinoptilolite additions improve the corrosion resistance of reinforcement at exposition long periods at chloride ions. This conduct can be attributed at the diminished of the pores size in the clinoptilolite structure, as consequence of the formation of the products originated during the pozzolanic reaction between the clinoptilolite with calcium hydroxide, carried out during curing time of mortar. As time advances, the pozzolanic reaction continues and the products of the reaction increases too, provoking the partial obstruction of the pores. Thereby the chloride ion penetrability capacity is diminished [33,34].

In the Figure 8 is compared  $E_{\text{corr}}$  vs  $i_{\text{corr}}$ , at one year of exposure with and without  $\text{Cl}^-$  at HR  $\sim 98\%$ . Can be seen that the values localized to lower than  $0.1 \mu\text{A}/\text{cm}^2$  with  $E_{\text{corr}}$  values more positive to  $-275 \text{ mV}$ , corresponding to mortars without exposure to ions  $\text{Cl}^-$ . The  $i_{\text{corr}}$  and  $E_{\text{corr}}$  values show a passivity state in the reference mortar and the mortars with zeolite. This behavior indicate that substitute until 25 % cement content by zeolite, not alter the high alkalinity conditions provided by the

cement, keeping passivity in the steel as indicated in the Pourbaix pH-potential diagrams. The most relevant at this time of the assay, is that the proposed mortars can improve the conditions of steel passivity, consequently, enhancing the durability of reinforcement concrete structures.



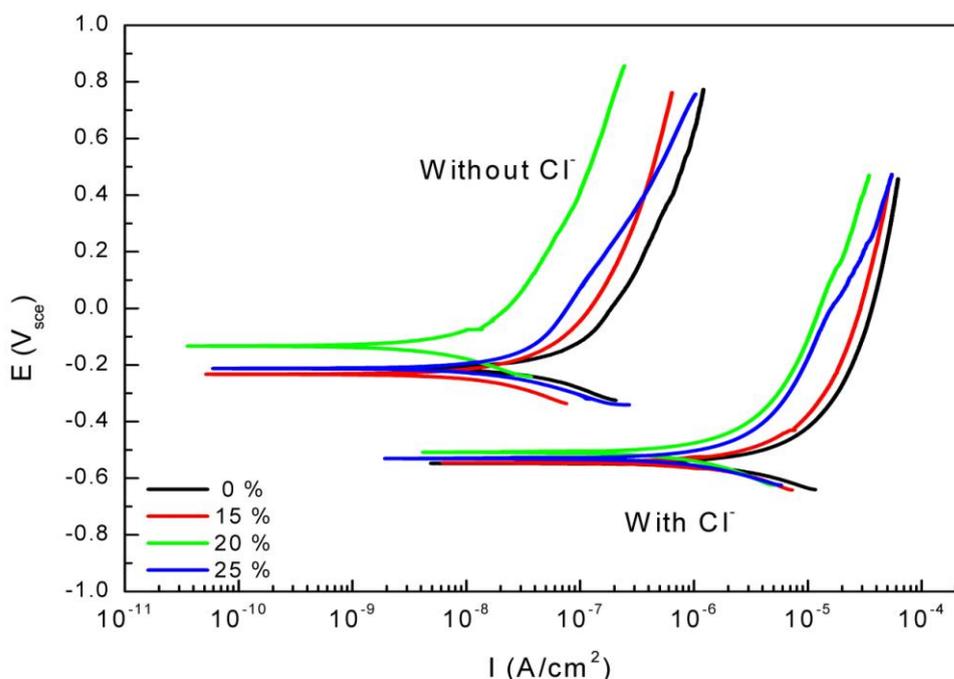
**Figure 7.**  $i_{corr}$  vs time of exposure of the steel bars embedded in mortar with  $Cl^-$  and different content of clinoptilolite.



**Figure 8.** Current density ( $i_{corr}$ ) versus corrosion potential ( $E_{corr}$ ) of the steel bars embedded in mortar without and with  $Cl^-$  with different content of clinoptilolite during one year of exposure in HR $\approx$  98%.

By other hand, the mortars exposed at aggressive medium (chloride ions) show  $E_{\text{corr}}$  values more negative than -275 and  $i_{\text{corr}}$  values between 3 and 9  $\mu\text{A}/\text{cm}^2$ , situated them in the high corrosion zone. With respect to the mortars containing zeolite, the  $E_{\text{corr}}$  values are localized at -450 to -550 mV and the  $i_{\text{corr}}$  values are between 0.3 and 9  $\mu\text{A}/\text{cm}^2$  covered very high and low corrosion zones, exhibited a better behavior than the mortar reference.

Figure 9 compares the polarization curves, obtained in the absence and presence of chlorides for the mortar reference and the mortars containing clinoptilolite at 365 days, after that the specimens were manufactured. Electrochemical parameters were calculated from this figure and are showed in Table 3.



**Figure 9.** Polarization curves of the steel bars embedded in mortar without (a) and with (b) (2%) of  $\text{Cl}^-$  with different contents of clinoptilolite.

Apparently, Figure 9 shows a significant gap in current densities -corresponding to the same values of electrical potential on the anodic portions of the curves- in presence and absence of chlorides ions. Is possible appreciate until two orders of magnitude, higher in mortars contaminated with chloride in comparison to mortars without chlorides, these discrepancies between these values were expected due to the presence and absence of the contaminate in the respective mortars .

The results of this figure are expressed more specifically after of calculate Tafel slopes showed in table 3. It can observe differences in corrosion rate, between the mortars containing zeolite and the reference mortars, mainly in the mortars contaminated with chloride. The reference mortar shows a value of 6  $\mu\text{A}/\text{cm}^2$ , while the mortars with zeolite, showed values near to 1  $\mu\text{A}/\text{cm}^2$ . The mortars containing clinoptilolite showed a better behavior against corrosion in both cases (with and without chlorides) than reference mortar. Although it is possible to observe a slight difference between the values of  $i_{\text{corr}}$ , the mortars containing 20% and 25% of clinoptilolite showed less corrosive activity values. These

values are in concordance with the results showed in Figure 4, 5, 6 and 7 obtained by other electrochemical technique.

**Table 3.** Corrosion current density ( $i_{\text{corr}}$ ) and Tafel pendants.

Clinoptilolite content in mortars (%)	$i_{\text{corr}}$ ( $\mu\text{A}/\text{cm}^2$ )	$\beta_A$ V/decade	$\beta_C$ V/decade
Without $\text{Cl}^-$ ions			
0	0.060	0.2414	0.1841
15	0.038	0.6119	0.181
20	0.016	0.1435	0.1289
25	0.023	0.9228	0.3563
With $\text{Cl}^-$ ions			
0	6.2	0.3959	0.2626
25	1.26	0.1813	0.1359
15	.710	0.0657	0.0582
20	1.01	0.1633	0.1469

#### 4. CONCLUSIONS

The principal contributions derivatives of compressive strength analysis, physical properties determination and corrosion assay of the mortars studied, are mentioned at following:

1. Compressive strength results showed that addition until 25% of clinoptilolite as replacement of the cement not declined the mechanical properties in the mortars; showing similar results to Portland cement mortars.

2. The decrease of the pore size in the mortars is attributed at the pozzolanic reaction carried out in mortars containing zeolite once that curing time has passed. The decrease of the pore size in the mortars creates a barrier effect to  $\text{Cl}^-$  ions, slowing down their arrival at the steel surface.

3. The additions of clinoptilolite until 25% as replacement of cement in the mortars not affect the passivity condition that supplies the high alkalinity of Portland cement.

4. In general, for all studied mortars the results show that the additions of clinoptilolite improved performance against corrosion of steel after one year of exposure in chloride ions presence.

5. Electrochemical studies were able to demonstrate the steel corrosive behavior embedded in mortars containing zeolites with and without expositions to an aggressive medium.

6. Encompassing all the results it can be said that replacement of the cement by clinoptilolite is a viable alternative to use as a construction material with a good mechanical resistance and good performance against corrosion in a contaminated medium with chlorides.

#### ACKNOWLEDGEMENT

The first author would like to thank to CONACYT for the scholarship with register number 63209. The authors express their gratitude to M.C. Martha A. Lomeli, for the technical assistance of electrochemical analyses.

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