

CORROSION RATES MEASUREMENTS BY LINEAR POLARISATION AND AC IMPEDANCE TECHNIQUES USING DIFFERENT STEEL BARS AND ACIDIC SOLUTION

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Abstrak

Laju korosi batang tulangan dalam beton dipengaruhi oleh tingkat keasaman atau tingkat pH dari lingkungan beton yang menyelimuti batang tulangan. Penelitian ini dilakukan dengan menggunakan larutan asam dengan derajat keasaman (pH) dan jenis asam yang berbeda serta jenis besi tulangan yang berbeda. Hasil penelitian ini menunjukkan bahwa laju korosi yang diperoleh dengan menggunakan kedua metode baik *Linear Polarisation Resistance (LPR)* maupun AC Impedance hampir sama. Hal ini disebabkan lingkungan asam mendukung proses korosi. Hasil penelitian ini juga menunjukkan bahwa jenis dan diameter batang tulangan serta konsentrasi larutan asam adalah faktor yang mempengaruhi laju korosi dari batang tulangan. Bahkan lama pengetesan juga mempengaruhi laju korosi karena tahanan polarisasi berkurang menurut waktu.

Keywords: laju korosi, konsentasi, larutan asam, tahanan polarisasi

Abstract

The corrosion rate of steel bars in concrete was affected by concentration of acid or pH level of concrete environment which covered the steel bars. This research was conducted by using different acid and concentration with different diameter and kind of steel bars. The results obtained from the test using both the polarisation resistance (LPR) and the AC impedance techniques are similar. This is because the acidic environment supports the corrosion process. It is also found that the type and diameter of bars immersed in acid solution and the concentration of acid are the determining parameters of the corrosion rates of the bars. Even the length of test period also affects the corrosion rates as the polarisation resistance decreases by time.

Kata kunci: Corrosion Rate, Concentration, Acid Solution and Polarization Resistance

1. Introduction

Corrosion is the deterioration of materials by chemical interaction with their environment. The term corrosion is sometimes also applied to the degradation of plastics, concrete and wood, but generally refers to metals. The most widely used metal is iron (usually as steel) and the following discussion is mainly related to its corrosion.

When steel reinforcement is encased in sound dense concrete, the entire surface of the steel is covered by a stable protective oxide film that forms in the alkaline environment created by

the hydration of the cement in the concrete. Under these circumstances no corrosion of the reinforcement can occur.

However, if the protective oxide film is locally destroyed, for example by the ingress of chloride ions, areas of different potential can be set up on the surface. The presence of acid affects the corrosion rates of steel bars in concrete. The steel bar is passive in a high pH environment (between 12 – 14) but the existence of acid in the concrete break down the pH of the concrete from the high level (alkaline

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environment) to the low level (acidic environment). Therefore, it is necessary to understand the behavior of the steel in acidic environment with different acids and various concentrations.

2. Literature Review

Concrete is a very durable material, which can be used for most types of construction. Its properties and performance are influenced by the selection of mix ingredients, mix design, placing, compaction, curing conditions, design and detailing, and interaction with service environment. The process of degradation, such as corrosion of steel reinforcement, is therefore dependent on concrete quality as well as exposure conditions. The initiation and propagation of corrosion in concrete structures can be influenced by both internal and external factors. These sources of deterioration depend on concrete properties and exposure conditions and, to a large extent, govern structural performance and remediation practices.

2.1 Overview of Concrete Deterioration Processes

While concrete has evolved to become the most widely used structural material in the world, the fact that its capacity for plastic deformation is essentially nil imposes major practical design limitations; this shortcoming is most commonly overcome incorporation of steel reinforcement into those locations in the concrete where tensile stresses are anticipated. regarding Consequently, concerns performance must not only focus upon properties of the concrete per se but also of the embedded steel and, in addition, the manner in which these two components interact.

In this regard, steel and concrete are in most aspects mutually compatible, as exemplified by the fact that the coefficient of thermal expansion for each is approximately the same. Also, while boldly exposed steel corrodes actively in most natural environments at a rate that requires use of extrinsic

corrosion control measures (for example, protective coatings for atmospheric exposures and cathodic protection in submerged and buried situations), the relatively high pH of concrete pore water (pH > 13.0-13.8) promotes formation of a protective passive film such that corrosion rate is negligible and decades of relatively low maintenance result.

2.2 Corrosion Basics

The surface of the corroding metal acts as a mixed electrode, upon which coupled anodic and cathodic reactions take place. At anodic sites, metal atoms pass into solution as positively charged ions (anodic oxidation) and the excess of electrons flow through the metal to cathodic sites where an electron acceptor dissolved oxygen is available consume them (cathodic reduction). This represents the electrochemical theory of metal corrosion; describing the metal corrosion process. combination of an anodic oxidation, such as metal dissolution, and a cathodic reduction, such as oxygen reduction or hydrogen evolution.

The electrons created in the anodic reaction must be consumed elsewhere on the steel surface establishing the corrosion reaction. The process is completed by the transport of ions through the aqueous phase, leading to the formation of corrosion products at the anodic sites either soluble (e.g. ferrous chloride) or insoluble (e.g. rust, hydrated ferric oxide).

If the current caused by the electron flow could be measured at all, the measured quantity, l_{net} would represent a net effect of the partial currents resulting from oxidation and reduction. l_{net} is generally zero, i.e. for the situation where a metal corrodes due to an oxidation reaction of the metal and one (O_2 -reduction) or two simultaneous reduction reactions (O_2 -reduction and O_2 - evolution) occurring on the same metal (Broomfield, 1997).

$$I_{net} = \sum I_{ox} - \sum |I_{red}|$$
(1)

The corrosion rate p is then proportional to the sum of the partial anodic currents (corrosion current) causing metal dissolution. p is defined as the loss of the corroding metal in micrometers per year [μ m/y] and can be calculated by (Andrade, 1996):

$$p = \frac{M i_{corr} t}{\rho z F} \qquad(2)$$

where,

M = atomic weight (= 55.85 g/mol for iron)

 $i_{corr} = \frac{Icorr}{A}$ (corrosion current density (A cm⁻²))

I_{corr} = corrosion current

A = measurement area

t = time

 ρ = density of iron (= 7.86 g/cm³)

z = number of electrons transferred per atom

 $(2 for Fe \rightarrow Fe + 2e^-)$

F = Faraday's constant (= 96500 C/mol)

This gives a conversion of $1\mu A = 11.6 \mu m$ steel section loss per year to obtain the rate of corrosion. The corrosion current that is inversely related to the polarization resistance can be calculated by the equation (Broomfield, 1993):

$$I_{corr} = \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c)} \frac{I}{R_p} = \frac{B}{R_p} \dots (3)$$

where β_{α} , β_{c} are the anodic and cathodic Tafel constant respectively, which is known as the Stern-Geary constant, B. B is taken as approximately 25 mV for actively corroding steel and around 50 mV for passive steel in concrete (Andrade, 1993). However, some sources took 26 mV and 52 mV (Millard, 1994) for actively and passive corroding respectively, with the error factor is 2.

Furthermore, guidance relating polarization resistance (Rp), corrosion

current density (*i_{corr}*) and corrosion penetration (p) to rates of corrosion is aiven in Table 1

A corrosion current density of 1 mA/m2 iron surface is therefore equal to a corrosion rate of 1.16 µm/year. If a rebar with a diameter of 16 mm is corroding with 100 mA/m2 surface for 20 years - which can locally be the case the cross section would have reduced to 11.4 mm. This can cause already static problems for the structure. In fact, the collapse of the Berlin Congress Hall and of a parking garage in Minnesota is two examples of spectacular failures because the static load capacity was reduced excessively due to corrosion (Borgard, B., 1990).

The electrochemical system "steel corroding in concrete" can be described by applying the mixed metal theory. The current density-potential curve can be achieved theoretically by solving the Butler Volmer equations in combination for the reactions that happened in anodic and cathodic.

In alkaline and oxygen rich electrolytes such as atmospherically exposed reinforced concrete structures, second and or the electrochemical reactions are involved in the overall corrosion reaction. If the Iron were just to dissolve in the pore water of the concrete, cracking and spalling of the concrete are not visible. Several more steps must occur for forming "rust". One combination is shown below where ferrous hydroxide and then hydrated Ferric oxide or rust (Broomfield, 1993):

$$Fe^{2+} + 2OH^{-} \rightarrow Fe(OH)_{2}$$
 ferrous hydroxide...(4)

$$4Fe(OH)_2 + O_2 + 2H_2O \rightarrow 4Fe(OH)_3$$
 ferric hydroxide...(5)

$$2 Fe(OH)_3 \rightarrow Fe_2O_3 . H_2O + 2 H_2O$$

hydrated ferric
oxide (rust)(6)

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Rate of	Polarization	Corrosion current	Corrosion
Corrosion	resistance: Rp	density: i _{corr}	penetration: p
COHOSION	$(k\Omega.cm^2)$	$(\mu A/cm^2)$	(µm/year)
High	2.5 > Rp > 0.25	$10 < i_{corr} < 100$	100 < p < 1000
Medium	25 > Rp > 2.5	$1 < i_{corr} < 10$	10 < p < 100
Low	250 > Rp > 25	$0.1 < i_{corr} < 1$	1 < p < 10
Passive	Rp > 250	i _{corr} < 0.1	P < 1

Source: Gowers and Millard, 1999

Unhydrated dense ferric oxide (Fe₂O₃) has a volume of about twice that of steel replaced. When it becomes hydrated it swells even more and becomes porous, increasing the volume at the steel/concrete interface two to ten times. This leads to the already mentioned cracking and spalling of the concrete observed as а consequence of steel corrosion in concrete. The electrochemical behavior of steel in aqueous solution has to be considered as the base understanding the complex corrosion process in the very inhomogeneous concrete with local gradients of pH and concentration of aggressive ions.

2.3. The effect of pH

The corrosion rate of active metals is strongly determined by the pH value and in neutral media by the oxygen content. Alkaline concrete has a pH value of about 12.5. In this environment carbon steel is passive and suffers therefore no noticeable corrosion in absence of chlorides. In neutral water the relatively slow diffusion of the oxygen to the metal surface is the limiting step in the corrosion process. The rate of corrosion of active metals in water caused by the O₂ - corrosion type is generally low and hardly exceeds 0.1 mm/year.

In macro cells there is a current flow which causes an additional metal dissolution at the anode. The current and thus the amount of material loss depends mainly on the difference of the corrosion potentials, the electrical resistance between anode and cathode, the ratio of the anodic and cathodic areas and the polarisation

behavior of the two metals. In practice typical corrosion rates are in the range between 0.5 and 2 mm/year [Bindschedler, D., 2001].

Also in the case of localised corrosion of passive materials the corrosion rates are usually very high. For pitting and crevice corrosion material losses up to 3 mm/year are not unusual. In the literature even corrosion rates of 20 mm/year are reported. Stress corrosion cracking and intergranular corrosion can, at least in unfavourable cases, lead to failures practically without preliminary warning.

It can already' be seen that the reduction of H^+ to H2 is a thermodynamically feasible way to allow the oxidation of Fe to take place. This also implies that (especially in deoxygenated solutions) the concentration of H^+ is very important.

Since pH = $-log [H^+]$, then: using the Nernst equation one can write:

$$E = E^{0} + \frac{2,3RT}{nF} \log a_{H^{+}} = E^{0} - 0,059pH$$
(7)

So, E for the hydrogen evolution (H+ reduction) changes by 59 mV for every change in pH unit.

Corrosion experiments intended to simulate steel in concrete have historically employed a saturated Ca(OH)₂ solution, the pH of which is approximately 12.4. However, with the advent of the pore water expression (12-14)method and theoretical considerations, it was recognized that K+ and Na+ are the predominant cations; and the solubility and concentration of these is such that a pH in excess of 13 typically occurs.

Limitations associated with pore water expression include, first, prior water saturation of samples is required and. second, the method is more useful for pastes and mortars since expression yields for concrete, particularly high performance ones, is low. Consequently, both ex-situ and in-situ leachina methods (Sagüés, A.A. et all, 1997) have also been developed, where the former involves exposure of a powder sample distilled water and the latter placement of a small quantity of water into a drilled cavity in hardened concrete. A limitation in the case of exsitu leaching is that solid Ca(OH)2 from the concrete becomes dissolved and elevates [OH-] compared to what otherwise would occur. Also, the dissolved Ca(OH)₂, if saturated, buffers the leachate at a pH of about 12.4. These limitations are minimized by the insitu method because only about 0.4 ml of distilled water is employed; however, water saturation of the specimen is required here also. Recently, modification of the exsitu method was proposed whereby a correction is made for the [OH-] resulting from Ca(OH)2 dissolution (Sagüés, A.A., et all, 1997); however, solubles in unreacted cement particles may also become dissolved. thereby elevating the calculated pH compared to what actually existed in the pore water. Consequently, this procedure may be more a measure of inherent alkalinity than of pore water pH.

2.4. Breakdown of the passivity due to pH-decrease

Passive hydrated oxides interact with the solution due to their certain solubility. If the solubility of the hydroxide (hydrated oxide) in a given aqueous environment is small then it is probable that it will form a stable protective film on the metal surface. However, the (hydrated) passivating oxide hydroxide films on many metal surfaces exhibit increasing solubility with decreasing pH of the surrounding solution.

Increasing solubility of the oxide layer will often imply a reduced passivity and an increase in the corrosion rate. Iron in aqueous electrolytes is passive when the hydroxide and oxide species $Fe(OH)_2$, Fe3O4 and Fe2O3 are stable. The regions where the soluble species Fe_2^+ and $HFeO_3^-$ are stable are the zones in which active corrosion is expected.

3. Experimental Program

The purpose is to evaluate the rate of corrosion of the steel bar and how this is affected by acid with various pH, bar with different diameters, type of steel bar, and time of length of exposure. The tests are performed by both the linear polarisation resistance (LPR) and AC impedance (EIS) techniques using acid solution as a medium and steel bars as the test specimen. The test results of the two techniques then are compared.

The reference electrode used was an Ag/AgCl of 3 % solution and the auxiliary electrode as well as the working electrodes comprised steel bars with 10 cm long. The working electrode were four different bars i.e. mild steel bar with diameter of 6 mm, 10mm, 25 mm and another one which is a stainless bar with a diameter of 10 mm. A 10 cm length of stainless steel with a diameter of 10 mm is used as the auxiliary electrode.

Furthermore, the experiment uses two kinds of acid i.e., acetic acid and sulphuric acid. The purpose of this is to compare the corrosion rates in the different acids. Both the acids used consist of three different pH's i.e., the value of 3, 4 and 5.

Similarly, Analogous to the Resistor-Capacitor Circuits test, the test is conducted by connecting reference electrode, working electrode and auxiliary electrode from the test specimen to the reference electrode, working electrode auxiliary and electrode from the ACM Field Machine as shown in Figure 18 below. The measurements of corrosion rates were performed after the steel bars were immersed for a day in the acid solution in a glass container.

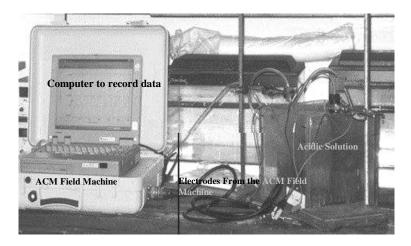


Figure 1. Connection between the electrodes from the ACM Machine and the electrode from Specimen

However, unlike the working electrode bars, once the tests were finished the auxiliary electrode was taken out from the specimen (aqueous solution) to keep its surface area as well as the reference electrode.

The acid solution was aerated continuously as long as the tests were performed by an electric driven air pump. This aim was to provide oxygen, which is needed, for the corrosion process. The electrode area was obtained from the surface area of the working electrode. The 'sequence' program was used to set up the tests of both the linear polarisation resistance and the AC impedance techniques providing a delay time between the two tests by means the 'pause' technique.

4. Results and discussion

The experiment of each concentration of both the acetic acid and sulphuric acid was performed for 7 days. In general, all of test results show that there is a rapid exponential decrease in the polarisation resistance until a certain time. There is then a slow decrease in the next time period as shown in Figure 2 below. The graph presents the test result of the polarisation resistance test using a mild steel bar of 6

mm diameter as a corrosion specimen in a acetic acid of pH 3.

From the results seen in Figure 2, it is seen that the polarisation resistance decreases with time for the mild steel specimen. As the corrosion current is inversely related to the polarisation resistance then it can be seen in Figure 3 that the corrosion rate increases in magnitude over 5 times during the 6 days test period. However, both the graphs show that after 5 to 6 days (on the end of test period) the rate of corrosion appears to stabilise.

The Figure 2 shows that the polarisation resistance of the 6 mm mild steel bar decreases for the first three days. It reduces to more than half of its value at the start of the test i.e., from the value of 418 Ω on the first day to 165 Ω on the third day (average results of the linear polarisation resistance and the AC impedance). The polarisation resistance then reduces more slowly between the third day and the sixth day of the test of the value of 165 Ω and 113 Ω respectively. Finally, the corrosion reaction looks to be constant at the last two days of the test i.e., from the value of 113 Ω on the sixth day to 111 Ω on the last day.

In contrast, the Figure 3 below show that there is a rapidly increase in

the corrosion current for the first three days from a value of 63 μ A/cm2 on the first day of the tests to a value of 125 μ A/cm² on the third day of the tests. This is almost twice the value of the result on the first day. The corrosion reaction then becomes more constant

on the last two days of test i.e., from a corrosion current value of 235 μ A/cm² to a value of 239 μ A/cm². Therefore, the rate of corrosion in the specimen will remain constant if the environment of the tests is not changed.

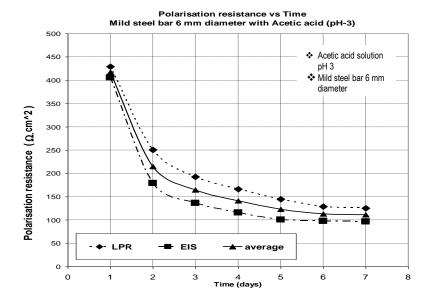


Figure 2. Plotting the polarisation resistance (Ω .cm²) vs. Time (days)

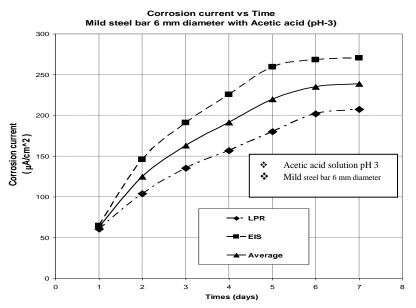


Figure 3. Plotting the Corrosion current (μ A/cm²) vs. Time (days)

Figure 4 below presents the result of the tests, which were performed using the same of acid and concentration with different diameter of the bars. It can be seen from the figure that the polarisation resistance of a stainless steel bar is higher than the polarisation resistance of a mild steel bar. A comparison the test results polarisation resistance shows that the polarisation resistance of the stainless steel bar is almost twenty times higher than the polarisation resistance of the mild steel bar i.e., 4062 Ω .cm² and 206 Ω .cm² respectively. The results shown are for the same 10 mm diameter of bar and for immersion in the same concentration of acid. This is because stainless steel bar has a passive oxide layer that acts as a corrosion inhibitor that protects the bar surface from corrosion.

The figure also shows that using a bigger diameter of the same type of bar i.e., mild steel gives a higher polarisation resistance in the beginning of test. However, at the end of the tests it is found that the results are just a little different but still show that the bigger diameters of the steel bar give the higher polarisation resistance. The results from first day of test using mild steel bars of 6 mm, 10 mm and 25 mm diameter gives the polarisation resistance results of 3246 Ω .cm², 746 Ω .cm², and 419 Ω .cm². And at the end of test the polarisation resistances of the bars are 206 Ω .cm². 150 Ω .cm², and 111 Ω .cm² receptively. Furthermore.

Polarisation resistance vs Time (average results of LPR and EIS Methods) Acetic acid (pH-3) with different bars

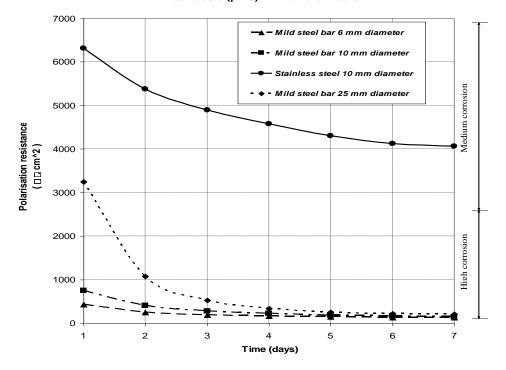


Figure 4. Measurement of polarisation resistance for different bar diameters with the same acidic solution

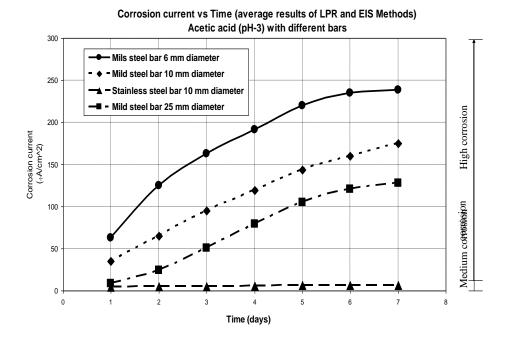


Figure 5. Measurement of corrosion current for different diameter of bars with the same acidic solution

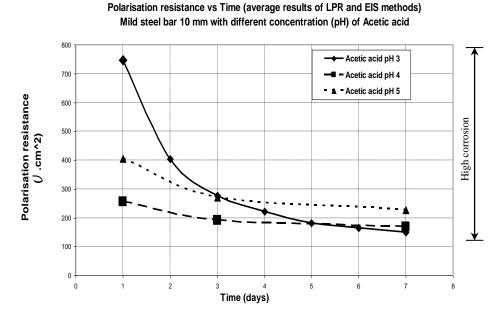


Figure 6. Polarisation resistance of the mild steel bar 10 mm diameter immersed in different concentration of acid

Another presentation of the test results can be seen in Figure 5 below. By plotting the current density against time, the figure shows that the corrosion current of stainless steel bar is quite constant. It is varies from a value of 4μ A/cm² at the beginning of the test to a value of 6.86 μ A/cm² at the end of the test, when the tests are performed for 7 days. However, the corrosion current of all the mild steel bars increased rapidly and exponentially over the same period. The corrosion current of the mild steel bars of the diameter 6 mm, 10 mm and 25 mm on the first day of testing were 64 μA/cm2, 37μA/cm2 and 10 μA/cm2 respectively. And the results on the end of the experiment are 271 µA/cm2, 190 μ A/cm2 and 140 μ A/cm2 respectively. The graph also shows that the corrosion reaction in the mild steel bars is slow on the fifth day and then looks quite constant on the last two days of the test. This suggests the equilibrium rate of the corrosion in each bar have been reached. Therefore, the corrosion rate reaches equilibrium if the environment is not changed.

Finally, Figure 6 below shows the effects of different concentrations of acid to the corrosion process of the steel bar. The figure shows the polarisation resistance results of a mild steel bar of 10 mm diameter.

The graph on figure 6 is displaced because the experiments use the same actual bars for the test of each concentration of acid. The first test uses the strongest acid i.e., acetic acid of pH 3, therefore, when the other tests using acetic acid of pH 4 and pH 5 the bar used already has a corroded surface. However, at the end of experiment it shows that the strongest acid i.e., acetic acid of pH 3 gives the lowest of the polarisation resistance results. It can be seen that the results of both the acetic acid of pH 4 and of pH 5 give a higher polarisation resistance result than using acetic acid of pH 3.

The polarisation resistance of bar in acetic acid of pH 3 decreases more dramatically than the other concentrations of acetic acid. This means that strong acids are more corrosive than weak acids. As the result of the strong acid more corrosion current can be passed. In other words, the use of strong acids will accelerate the corrosion reaction.

Therefore, the lower pH of an acid solution used in the experiment the higher rates of corrosion can be obtained. The corrosion process is faster with the lower pH of acid solution rather than with the higher pH of acid as presented in Graph 10 above. It also seems that the corrosion current for both of the two different pH i.e., pH 4 and pH 5 increases more slowly than the corrosion current with the acetic acid solution of pH 3.

The corrosion current for the tests, which used acetic acid solution of pH 4 and pH 5, are from $103.67\mu\text{A/cm}^2$ to $155.82\mu\text{A/cm}^2$ and from $64.38~\mu\text{A/cm}^2$ to $115~\mu\text{A/cm}^2$ respectively. The corrosion current for the experiment used the acetic acid solution of pH 3 increased from $35.07\mu\text{A/cm}^2$ to $175.09\mu\text{A/cm}^2$. Comparing the total increasing of the corrosion current for each concentration of the acetic acid solution shows that the biggest increase of the corrosion current is in the acetic acid solution of pH 3, which is the strongest of the acids.

5. Conclusion

1. The results of the experiment which used the mild steel bars show that the corrosion rates of the mild steel bars which were immersed in acid are very high corrosion in which each the mild steel bars have a value of the corrosion current over of 100 µA/cm². While the results of the experiment, which used the stainless bar, show that the corrosion rate of a stainless bar, which was immersed in the same acid and concentration with the mild steel bars, is lower than corrosion current of the mild steel bars. The corrosion rate of the stainless bar is expected to be a passive corrosion, however, as it was immersed in a strong acid solution with a value of pH 3, which broke down the passive layer of the stainless bar. The stainless

- bar then corroded which is categorised in a medium corrosion with the corrosion current below of 10 μ A/cm².
- 2. The results of the corrosion rate measurement, which used an acid solution, show that both the linear polarisation resistance (LPR) and the AC impedance techniques give similar results. The analogous resistor-capacitor circuit tests have been performed to measure the polarisation resistance Rp using both the linear polarisation resistance (LPR) and the AC impedance techniques by means the ACM Field machine.
- 3. The obtained results are similar to the expecting result, before performed the tests with various variables such as using different concentration of the acid, different diameter and type of the bars particularly for the mild steel bars. There is little bit different from the expecting results for the stainless bar, which is expected to be a passive corrosion level, however, the results show that the bar is in the medium corrosion level. This because the acid used was strong. However. the results are reasonable to be good results because the surface of the stainless bar was looked much damaged after performed the experiment.

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