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# Influence of polyvinylpyrrolidone on rebar corrosion in sulphate solution

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**Abstract** This paper reports the results of an experimental investigation of the polyvinylpyrrolidone (PVP) influence on the steel reinforcement corrosion and compressive strength of concretes in sulphate medium. The effect of admixture of PVP in concrete on the corrosion resistance of steel reinforced concrete was assessed by measuring electrochemical test during 60 days immersion in two different external solutions. AC impedance spectrum indicated that the resistance of PVP mixed electrodes were higher than those without PVP. The compressive strength of concrete specimens containing PVP was measured and an increase of 19%~24% was observed.

**Keywords :** reinforcing steel; corrosion; polyvinylpyrrolidone; AC impedance; sulphate.

## 1. Introduction

It is generally agreed that embedded steel rebars in concrete are not corroded under ideal conditions because of the alkaline medium present which provides a protective environment for the steel reinforcement (Chen 1998, Batis *et al*. 2000). The long-term durability of this protection against corrosion is connected with the stability of the necessary conditions of the passive layer. However, if the passive film in the concrete is broken down either by carbonation, or by the presence of corrosive ions such as chloride, $SO_2$ or $NO_x$, the accumulation of corrosion products such as iron oxides or hydroxides causes an expansion and interior micro-crack within the concrete which in turn leads to a direct contact between the steel and the electrolyte (Assaad *et al*. 2005, Al-Dulaijan *et al*. 2003, Turkmen and Gavgali 2003). Similarly, sulphate ions from external source attack the concrete structures and deteriorate the reinforced concrete. They also can reacts with $C_3A$ in the cement to form Ettringite and since the reaction products occupy a higher volume, internal stresses are generated thus to disrupt the concrete (Assaad *et al*. 2005, Yilmaz *et al*. 1997, Ramyar and Inan 2007, Vu and Stewart 2000, Loto 1992).

Corrosion can be controlled by various methods such as the variation of the concrete formulation, cathodic protection, enhancing the performance of concrete through the application of surface treatments

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(hydrophobic agents or surface coatings) and the use of chemical inhibitors (Al-Mehthel *et al.* 2008, Söylev and Richardson 2008). Corrosion inhibitors have long been considered as one of the most effective solutions of the corrosion problem of steel reinforcement in concrete structures. The addition of an inhibitor to the mixing water is one of the more convenient, attractive and inexpensive process for preventing corrosion of the embedded steel when added in an adequate amount (Söylev and Richardson 2008). Therefore, they offer protection by forming a protective film at the steel surface and reducing the ingress of aggressive ions into the concrete matrix (Batis *et al.* 2001, Hou and Chung 2000, Monticelli *et al.* 2000). Corrosion inhibitors are either added to concrete during its preparation or penetrated into the hardened concrete surface. Organic and polymer based substances such as amines, alkanolamines, amine salts, emulsified mixtures of esters, alcohols and amines have been used as corrosion inhibitors for the protection of rebar in concrete. Organic inhibitors work by forming a monomolecular film between the metal and the solution (Gaidis 2004, El-Shafei *et al.* 2001, Sadegzadeh *et al.* 1993, Gürten *et al.* 2005, Gürten *et al.* 2007, Nmai *et al.* 1992). These compounds block capillary channels and pores in concrete and diffusion becomes more difficult and thus diffuse layer resistance increases. Several polymers have been also used as inhibitor in concrete and have been adsorbed on the surface of the metal to protect it against the corrosive environment.

In this work, we aimed to study the effectiveness of polyvinylpyrrolidone (PVP) on the corrosion of the reinforced steel in 0.1 M $Na_2SO_4$ solution. As mentioned above, the formation of Ettringite is mainly responsible for deterioration of the concrete matrix. Therefore, we have investigated the PVP effect on the sulphate ion mobility from the external solution to metal surface through micro cracks in deteriorated concrete and determined the effect of the inhibitor on the compressive strength of concrete. Corrosion potential ($E_{corr}$) monitoring and electrochemical impedance spectroscopies (EIS) were the techniques chosen for this study. These techniques have been extensively used in laboratory investigations of the corrosion behaviour of reinforced concrete. EIS was found to have the major advantage of providing the possibility of establishing a physical interpretation of the processes involved (Carnot *et al.* 2003).

## 2. Experimental

### 2.1. Materials and specimen preparation procedures

Steel bars used for reinforcement had the chemical composition of 0.13% C, 0.65% Mn, 0.030% S, 0.013% P and 0.017% Si. 50 mm long test electrodes with 17.27 $cm^2$ contact area and 10 mm diameter were mechanically polished by using 600~1200 grades wet SiC papers. Reinforced concrete specimens were prepared using a cement type II/B (P-L) 32.5-R. The chemical composition of cement used in experiments is given Table 1. Natural river aggregate was used in experiments. The coarse and fine aggregates were washed to remove salts, dust and other fine particles. The composition of the concrete is given in Table 2. Saturated dry surface density of aggregate was $2.59 \times 10^3$ $kg.m^{-3}$ and their sizes ranged from 0.5~4 to 4~8 mm. Cylindrical reinforced concrete specimens of 70 mm long and 40 mm in diameter with embedded reinforcing steel bar were prepared for electrochemical studies and were connected by means of copper wires. The water/cement (w/c) ratio was 0.5. The electrodes were post-cured for 24 h and then were placed in the external solution. Concrete specimens were divided into two groups. The first series were prepared using distilled water (specimen A-C) whereas the second was mixed with sulphate solution (specimen A'-C'). The concrete specimens were placed in 0.1 M $Na_2SO_4$ solution. PVP inhibitor (Merck GR grade) was added to the mixing water in the specimen B and B',

Table 1 Chemical composition of cement

| $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ |
|---|---|---|---|---|---|
| 28.98 | 7.80 | 3.61 | 48.00 | 2.17 | 2.38 |

Table 2 Composition of the concrete

|  | Mass (kg) | Density (kg/$dm^3$) | Volume ($dm^3$) |
|---|---|---|---|
| **Water** | 180.00 | 1.00 | 180.00 |
| **Cement** | 360.00 | 2.96 | 120.96 |
| **Aggregate** | 1731.10 | 2.59 | 668.38 |
| **Air** | - | - | 30.00 |

and in the exposure solution of specimen C and C'. Detailed information about the external and mixing water environments are given in Table 3. All types of tests namely; corrosion potential, polarisation resistance, AC impedance and compressive strength were carried out on three individual samples from each specimen.

### 2.2. Test methods

Impedance measurements were carried out at the open circuit potential ($E_{oc}$), using a computer-controlled electrochemical analyzer CHI model 604 A and frequency response analyser and the three-electrode assembly. Prior to the impedance measurement, a stabilisation period of 30 min was allowed, which proved to be sufficient to attain a stable value for $E_{oc}$. The measurements carried out with applied sinusoidal potential waves of 5 mV amplitudes around corrosion potential, with frequencies ranging from $10^5$ Hz to $10^{-1}$ Hz. The AC impedance results were fitted by using the Zview software from Scribner Associates. The measurements were carried out on the 1, 2, 7, 28 and 60th day of the curing period (Wang *et al.* 1998, Jamil *et al.* 2005).

The detection of corrosion by using potential measurements, provide information on the time to initiation of reinforcement corrosion under laboratory conditions, is one of the most typical procedures for the routine inspection of reinforced concrete structures. Corrosion of the specimens was monitored by measuring the corrosion potentials and recorded at regular intervals, until equilibrium conditions were established, using multimeter versus a SCE for a period of 60 days (Pech-Canul and Castro 2002, Batis *et al.* 2001).

In compressive experiments, cubic shaped concrete specimens (15 cm × 15 cm × 15 cm) were used. The

Table 3 External solution and mixing water environments*

| Specimens | External solution | Mixing solution |
|---|---|---|
| **0** | Distilled water | Distilled water |
| **A** | 0.1 M $Na_2SO_4$ | Distilled water |
| **B** | 0.1 M $Na_2SO_4$ | Distilled water + 100 ppm PVP |
| **C** | 0.1 M $Na_2SO_4$ + 100 ppm PVP | Distilled water |
| **A'** | 0.1 M $Na_2SO_4$ | 0.1 M $Na_2SO_4$ |
| **B'** | 0.1 M $Na_2SO_4$ | 0.1 M $Na_2SO_4$ + 100 ppm PVP |
| **C'** | 0.1 M $Na_2SO_4$ + 100 ppm PVP | 0.1 M $Na_2SO_4$ |

*Values are based on triplicate analysis.

compressive strength was determined after curing for 28 days in external solution (0.1 M $Na_2SO_4$, 0.1 M $Na_2SO_4$ + 100 ppm PVP or distilled water) by using Tonitecnic compressive testing machine. The average compressive strength of the three concrete cubes was determined by loading at a constant rate of 4~5 kN/s using a hydraulically operated digital compression machine of 3000 kN capacity according to ASTM C 39 (Al-Amoudi *et al.* 2003). The degree of deterioration was also evaluated by the following equation:

$$\% = [(A-B) / A] \times 100$$

Where A is the average compressive strength of three specimens cured under distilled water, MPa; and B is the average compressive strength of three specimens exposed to the test solutions, MPa.

## 3. Results and Discussions

### 3.1. Corrosion potential ($E_{cor}$) of embedded steel

Fig. 1 presents the variation of the rebar corrosion potential in time for the specimens (blank and treated with PVP) exposed to sulphate solution. $E_{cor}$ for the specimens in all media have varied from −0.100 V to −0.578 V (vs SCE) for a period of 60 days. These values are the averages of three measurements and the standard deviations were found to be in the range 0.023~0.032 for n = 3.

$E_{cor}$ values in the concrete specimen without corrosion inhibitor (specimen A) was more negative than the corrosion potentials of the steel in the concrete specimen prepared with PVP (specimen B) and the specimen prepared with distilled water which was cured in 0.1 M $Na_2SO_4$ containing 100 ppm PVP external solution (Specimen C) as indicated in Fig. 1a. All specimens (Specimen A-C) were more positive than the ASTM C 876 threshold value of −270 mV SCE (saturated calomel electrode) after 8 days of exposure (ASTM C 876).

Corrosion potentials on steel bars in the concrete specimen prepared with 0.1 M $Na_2SO_4$ and which was cured in 0.1 M $Na_2SO_4$ external solution (Specimen A') and the specimen prepared with 0.1 M $Na_2SO_4$, cured in 0.1 M $Na_2SO_4$ containing 100 ppm PVP external solution (specimen C') were more negative than the potentials of the concrete specimen prepared with 0.1 M $Na_2SO_4$ incorporating PVP

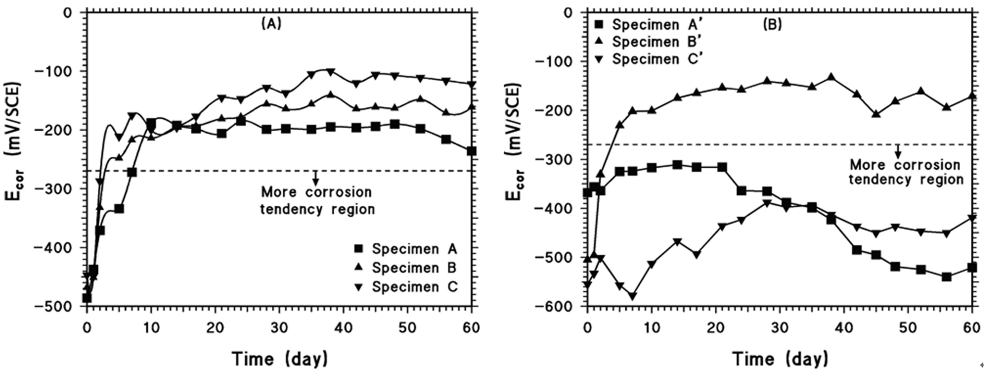

Fig. 1 Potential-time records for the various reinforced concrete specimens in the sulphate and sulphate containing PVP environments

(specimen B') (see Fig. 1b). The corrosion potentials on steel in the concrete specimen prepared with PVP (Specimen B') were more positive than –270 mV SCE after 3 days of exposure, indicating the passivity of reinforcing steel during 60 days of exposure. According to ASTM C 876, potentials more negative than –270 mV, with respect to SCE, indicate greater than 90 % probability of active reinforcement corrosion. Values less negative than –120 mV SCE indicate a probability of corrosion below 5%, while those falling between –270 and –120 mV SCE indicate uncertainty of corrosion (ASTM C 876).

These data indicate that concrete specimen prepared with 0.1 M $Na_2SO_4$ containing PVP was efficient in delaying reinforcement corrosion in the concrete specimens in sulphate solution. In other words, while the addition of PVP with sulphate greatly reduced the corrosion probability, whereas specimen prepared with 0.1 M $Na_2SO_4$ (Specimen A') and concrete containing 0.1 M $Na_2SO_4$ as a mixing solution and PVP in added to the external solution (Specimen C') couldn't be more effective on corrosion resistance of rebar in concrete. It is seen that the specimens containing sulphate ions were more corrosive than the specimens without sulphate just as expected.

Sulphate ions from external sources do not cause deterioration of reinforced concrete structures for being prepared with distilled water (Specimen A-C). As expected, there would not be any destructive effect of sulphate ions which leads to the deterioration of concrete structure (Fig. 1a). Specimen prepared with 0.1 M $Na_2SO_4$ (Specimen A') caused micro cracks in concrete, which led to more serious deterioration. However, specimen containing PVP (Specimen B') can be restrained their corrosion attack on metallic material. Due to the fact that PVP is believed to block these cracks, and thus sulphate diffusion becomes more difficult to the reinforced steel's surface (Fig. 1b).

The corrosion potential provides only qualitative information about reinforcement corrosion. Reliable information on reinforcement corrosion can be entirely obtained by measuring the resistance or current density (Huang *et al.* 1996).

### 3.2. AC impedance method

The impedance measurements on steel bar in the concrete samples were carried out using platinum electrode as a counter electrode, SCE as a reference electrode and the steel bar embedded concrete as the working electrode (frequency range $10^5$ Hz to 0.1 Hz). The impedance parameters, solution resistance ($R_s$), pore resistance ($R_{po}$) include charge transfer resistance ($R_{ct}$) and diffuse layer resistance ($R_d$), differential pore capacitance ($Q_{po}$) and surface inhomogeneity coefficient (n) values are given in Table 4.

Fig. 2 shows the typical AC impedance behaviour observed up to 60 days exposure in all concrete specimens. As shown in Fig. 2a, (Specimen A) the diagram exhibits distorted semicircular change representing $R_s + R_{ct} + R_d$ until reaching the real impedance value 47.9 Ω (1.81 kHz) during the first day in Table 4. Resistance values were written in the Tables and Figures are given in the SI units in ohms. In this table, the frequency values at the beginning of Warburg region are also given. This region, which is known as distorted semicircle, can attain only one section because the surface structure of the steel is not disturbed at the applied maximum frequency. The solution resistance ($R_s$) can be obtained by extrapolating the distorted semicircle curve to the real impedance axis (Z'/ohm). The region of the real impedance and the imaginary impedance, which increases continuously, is known as Warburg impedance. This resistance that is due to coating, accumulating of surface and concrete, causes shielding effect or resistance effect for the metal corrosion (Gürten *et al.* 2005, Erbil and Doğa 1987, Dehri and Erbil 2000). The partially seen semicircular region of high frequencies included information the corrosion process occurring along the pores of concrete. Thereby, the diameter of this curve was equal to the pore resistance ($R_{po}$) which included the charge transfer resistance ($R_{ct}$) and diffuse layer resistance ($R_d$). $R_{ct}$ and $R_d$ are

Table 4 Determined values for the equivalent circuit of Fig. 3 obtained from the fitting procedure.

| **Specimen** | ***Time*** **(*day*)** | **$R_s + R_{ct} + R_d$ (Ω)** | ***Frequency*** **(*kHz*)** | **$Q_{po}\ s^n\Omega^{-1}$** | **n** |
|---|---|---|---|---|---|
| **A** | 1 | 47.9 | 1.81 | $3.85 \times 10^{-8}$ | 0.956 |
| | 2 | 60.8 | 5.44 | $1.35 \times 10^{-8}$ | 0.987 |
| | 7 | 147.1 | 1.81 | $1.50 \times 10^{-8}$ | 0.930 |
| | 28 | 321.9 | 0.83 | $4.46 \times 10^{-9}$ | 0.996 |
| | 60 | 780.9 | 0.22 | $1.24 \times 10^{-7}$ | 0.661 |
| **B** | 1 | 71.3 | 1.22 | $1.29 \times 10^{-8}$ | 0.960 |
| | 2 | 90.3 | 0.69 | $3.10 \times 10^{-8}$ | 0.892 |
| | 7 | 226.8 | 12.25 | $8.03 \times 10^{-9}$ | 0.961 |
| | 28 | 399.7 | 3.21 | $4.37 \times 10^{-9}$ | 0.989 |
| | 60 | 1073.7 | - | $5.11 \times 10^{-6}$ | 0.736 |
| **C** | 1 | 49.5 | 2.60 | $2.99 \times 10^{-8}$ | 0.970 |
| | 2 | 65.7 | 1.81 | $1.16 \times 10^{-8}$ | 0.987 |
| | 7 | 172.6 | 1.23 | $1.96 \times 10^{-8}$ | 0.897 |
| | 28 | 378.7 | 1.23 | $6.29 \times 10^{-9}$ | 0.988 |
| | 60 | 1016.8 | 0.01 | $2.66 \times 10^{-6}$ | 0.674 |
| **A'** | 1 | 41.1 | 9.69 | $3.85 \times 10^{-8}$ | 0.962 |
| | 2 | 54.4 | 6.94 | $1.74 \times 10^{-8}$ | 0.988 |
| | 7 | 149.7 | 1.23 | $2.17 \times 10^{-8}$ | 0.901 |
| | 28 | 292.4 | 9.62 | $5.96 \times 10^{-9}$ | 0.995 |
| | 60 | 785.7 | - | $1.71 \times 10^{-5}$ | 0.724 |
| **B'** | 1 | 52.8 | 4.63 | $4.68 \times 10^{-8}$ | 0.932 |
| | 2 | 63.5 | 1.81 | $5.26 \times 10^{-8}$ | 0.911 |
| | 7 | 177.3 | 2.60 | $1.70 \times 10^{-8}$ | 0.911 |
| | 28 | 546.4 | 0.46 | $3.21 \times 10^{-9}$ | 0.996 |
| | 60 | 864.9 | 2.00 | $1.36 \times 10^{-5}$ | 0.706 |
| **C'** | 1 | 61.6 | 3.21 | $1.69 \times 10^{-8}$ | 0.985 |
| | 2 | 86.1 | 1.81 | $1.16 \times 10^{-8}$ | 0.949 |
| | 7 | 209.6 | 3.68 | $9.44 \times 10^{-9}$ | 0.956 |
| | 28 | 579.5 | 0.46 | $4.06 \times 10^{-9}$ | 0.972 |
| | 60 | 1098.0 | 0.01 | $3.82 \times 10^{-6}$ | 0.603 |

directly related the rate determining resistance and permeability (porosity) of the oxide film at the reinforcing steel corrosion respectively. Identical results were reported by Tüken *et al.*(2007) in a study of the mild steel protection with polymer coating.

During the first day, The sum of $R_s + R_{ct} + R_d$ value for specimen A and Specimen C which were made of steel embedded in concrete containing only distilled water as a mixing solution, 0.1 M $Na_2SO_4$ and 0.1 M $Na_2SO_4$ + 100 ppm PVP respectively as external solution, were 47.9 Ω and 49.5 Ω respectively. The resistance value for specimens B containing distilled water and PVP as a mixing solution, 0.1 M $Na_2SO_4$ as external solution has increased to 71.3 Ω. However, the resistance of the specimen A' containing 0.1 M $Na_2SO_4$ as mixing solution, was measured as 41.1 Ω, this value has increased to 52.6 Ω and 61.6 Ω for specimens B' and C', respectively. These values indicate that specimen A' had already suffered from disruption of concrete by reason of Ettringite salts and that the addition of PVP to the concrete, has formed a protective layer on the steel and concrete surface. However, when the electrodes (specimen A-C') were kept in the external solution, for the longer periods the resistance values have increased gradually.

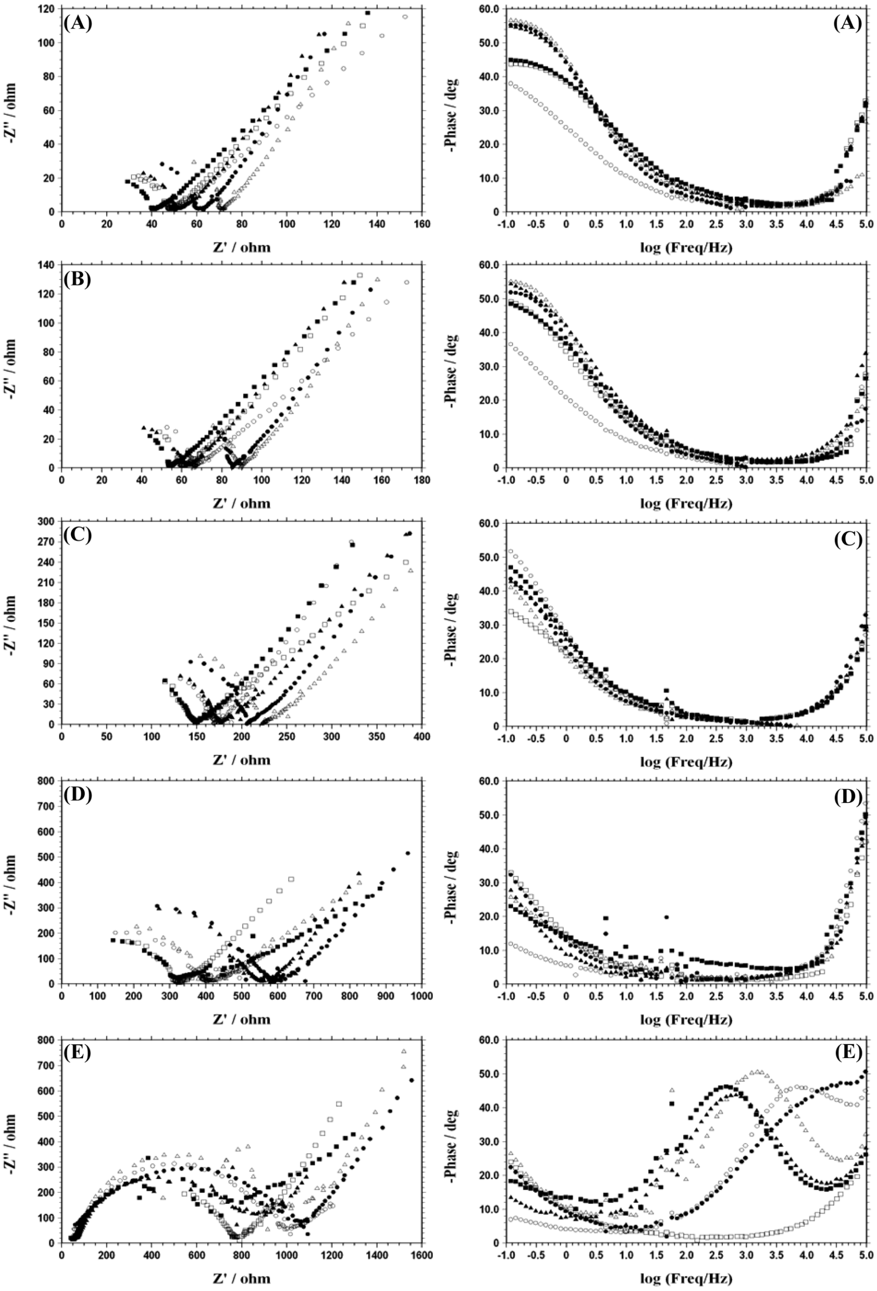

Fig. 2 Electrochemical impedance spectroscopy diagrams (Nyquist and phase angle Bode plots) of reinforcing steel [time (day) (A) 1 (B) 2 (C) 7 (D) 28 (E) 60] □ Specimen A, ■ Specimen A', △ Specimen B, ▲ Specimen B', O Specimen C, ● Specimen C'] In all of the diagrams, the maximum frequency in $10^5$ Hz and the minimum frequency values that correspond to Warburg region are given in Table 4. Explanations of legends are given in Table 3.

These increments in the resistance are due to the diffuse layer effect, and the $R_d$ resistance addition to the $R_{ct} + R_s$. After 60 days, the values of $R_s + R_{ct} + R_d$ have showed an increment from 71.3 Ω to

1073.7 Ω for specimen B and from 49.5 Ω to 1016.8 Ω for specimen C. Electrodes containing sulphate and PVP in their mixing and external solutions (specimen B' and C') have showed similar increment in resistances as 52.8 Ω to 864.9 Ω and 61.6 Ω to 1098.0 Ω, respectively.

In all media, Warburg impedance region has showed similar changes. Where, it is related to ion diffusion processes taking place through the pores of the concrete (covered surface) towards the underlying metal surface at metal/solution interface such a protective coating (Ozyılmaz *et al.* 2006). The rebar present in reinforced concrete has a pore structure where the capillary channels are in contact with external medium. When these capillary channels approach the metal ends, electrical double layer forms thus allowing charge transfer in this layer. Charge transfer resistance $R_{ct}$, diffuse layer resistance $R_d$ was called as a pore resistance ($R_{po}$) ($R_{po} = R_{ct} + R_d$) and differential pore capacitance (constant phase element) $Q_{po}$ are shown in Fig. 3. The outside concrete resistance ($R_{con}$) and the proposed differential capacitance for the concrete ($Q_{con}$) were fixed onto the circuit. The outermost double layer formed within the capillary channels and the solution resistance ($R_s$) that arose between the outermost of the concrete and the potential point measurement is also shown in Fig. 3. Pore resistance, differential capacitance and surface inhomogeneity coefficient (n) values were calculated using the Zview software from complex plane data. According to the equivalent circuit; high frequency region of Nyquist diagram shows the charge transfer resistance and diffuse layer resistance. However, increasing imaginary impedance in the lower frequency region is also considered as Warburg impedance (Fig. 3). These impedances are evaluated as a total resistances produced from both the concrete and corrosion products.

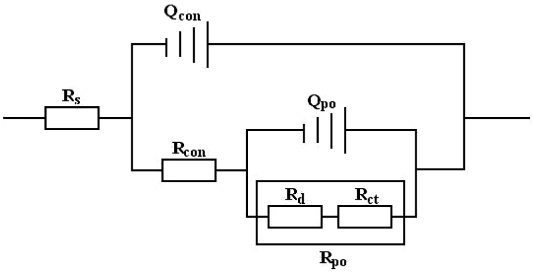

Fig. 3 The proposed equivalent electrical circuit model for concrete. $R_s$: Solution resistance, $Q_{con}$: concrete differential capacitance, $R_{con}$: concrete resistance, $Q_{po}$: differential pore capacitance, $R_{po}$: pore resistance $R_{ct}$: charge transfer resistance, $R_d$: diffuse layer resistance.

In our previous study, we have reported that when additives are added into mixing solution, molecules losses their mobility in a chloride medium, and thus the effect will be much less than that in the external solution (Gürten *et al.* 2005, 2007). However, in the sulphate medium, capillary channels, pores and internal stress in concrete are formed by Ettringite salt caused by the presence of the sulphate ions may lead to an increase in the mobility of the additive added to the mixing solution that affects the corrosion at the metal surface and its surroundings. However, at the present, it is difficult to impose this vision even though our experimental results support it.

### *3.3. Compressive strength*

The compressive strength values of the specimens that have been cured for 28 day are given in Table 5. These were 18.1~23.9 MPa. The lowest value belonging to the specimen cured in both mixing and external

Table 5 Compressive strength of the concrete specimens at 28 days*

| Test Series | Compressive strength (MPa) |
|---|---|
| **Specimen 0** | 19.4 |
| **Specimen A** | 21.9 |
| **Specimen B** | 23.9 |
| **Specimen C** | 22.1 |
| **Specimen A'** | 18.1 |
| **Specimen B'** | 22.4 |
| **Specimen C'** | 20.3 |

*Values are based on triplicate analysis with a maximum standard deviation of 0.5

solution in 0.1 M $Na_2SO_4$ was 18.1 MPa and the highest one to the specimen B was 23.9 MPa. The compressive strength of the concrete containing distilled water as mixing and external solution was observed at 19.4 MPa (Specimen 0), while the sample containing distilled water only as mixing and sulphate as external solution was found to be 21.9 MPa (specimen A). When PVP is added to the mixing solution, the compressive strength of specimen B was 24% and 19% more resistant than those cured in 0.1 M $Na_2SO_4$ as mixing and external solution and specimen containing distilled water as mixing and external solution respectively. The formation of Ettringite is mainly responsible for the large volume expansion during the maturation of concrete which leads to an increase in internal tensile stress and deterioration of the concrete matrix (Yilmaz *et al.* 1997). The results show that the PVP added to the mixing solution increases compressive strength in the ratio of % 24.

## 4. Conclusion

The investigation was carried out to examine the effectiveness of polyviniylpyrrolidone in diminishing corrosion of steel embedded in concrete specimens prepared with or without sulphate added to mixing or external solution. The following conclusions can be drawn from the investigation:

- Corrosion potential measurements revealed that specimens prepared with distilled water (specimen A-C) and the specimen prepared with 0.1 M $Na_2SO_4$ containing PVP are found to performed better than specimens prepared with 0.1 M $Na_2SO_4$ (specimen A' and specimen C').
- PVP improved the corrosion resistance of concrete when added to mixing or external solution mainly due to the increase of the diffuse layer resistance. The increase of the total resistances ($R_s + R_{ct} + R_d$) in the Nyquist diagrams and the decrease in shift of Warburg region to low frequencies supports the increment of the diffuse layer resistance and the diffusion occurred more difficult.
- Compressive strength measurements revealed that PVP added to the mixing solution of concrete increases compressive strength in the ratio of % 24

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