Corrosion-Resistance Characteristics of Concrete Containing Furfural

Dr. Suhair kadhem Al-Hubboubi ,Prof. Dr.Zain Al-Abideen Raouf & Dr.Ra' id Hasan Abbood

Abstract

Corrosion inhibiting admixtures are unique among other methods to protect reinforced concrete from corrosion damage. In this study, the effect of furfural on the fresh and hardened properties of concrete mixes of 35 and 45 MPa compressive strengths as well as the corrosion inhibition of furfural was evaluated. Furfural was added at different dosages (1, 2 and 3% by weight of cement) with and without superplasticizer (HRWR). Different electrochemical measurements were performed (Half-cell potential, Tafel plot and linear polarization resistance). Electrochemical measurements confirmed that furfural dramatically reduces the rate of corrosion; the inhibition efficiencies were 62.7 and 63.8 % due to 3% furfural addition to 35 and 45MPa-concretes respectively. Results also showed that the combined presence of furfural and HRWR was found to provide an excellent corrosion protection to steel.

الخلاصة

تعد مثبطات التآكل من الطرائق المهمة لحماية حديد التسليح من خطر التآكل.في هذه الدراسة تم تقييم تأثير الفورفورال على الخواص الطرية والمتصلبة لخلطات خرسانية ذات مقاومة انضغاط 35 و45 ميكاباسكال بالاضافة الى تقييم الفورفورال كمثبط لتآكل حديد التسليح .اضيف الفورفورال بعدة وزمات (2,1 و3%من وزن السمنت) مع وبدون الملدن المتفوق .اجريت عدة فحوص كهروكيميائية تضمنت (جهد نصف الخلية،بيانات تافل ومقاومة الاستقطاب الخطي).بينت القياسات الكهروكيميائية ان استخدام الفورفورال ادى الى تقليل كبير للتآكل وان كفاءة التثبيط كانت 62.7 و63.8% نتيجة اضافة 3% للخرسانة ذات المقاومة 35 و45 ميكاباسكال على التوالي وبينت النتائج ايضا ان الاستخدام المشترك للفورفورال والمضاف الملدن المتفوق وفرت حماية ممتازة لحديد التسليح.

Keywords: Corrosion inhibitor, Furfural, Superplasticizer, Linear polarization resistance.

1.Introduction

Concrete construction in Arabian Gulf seaboard countries shows an alarming degree of deterioration within the short span of 10 to 15 years. The deterioration is accentuated by the geomorphic and climatic environmental conditions which are characterized by reactive and marginal aggregates, high temperature –humidity regimes, and severe ground and ambient salinity **(Rasheeduzzafar, 1990)**. Several solutions to this problem have been proposed and tested, though to date no ideal solution has been found. The high costs or lack of effectiveness for some of the other solutions reveal several advantages for the utilization of corrosion inhibiting admixtures. Some of the admixtures, however, may retard time of setting of the cement or be detrimental at later ages. Many would be subject to leaching and hence less effective in concrete that has lost soluble material by leaching **(ACI 222R-96)**. Studies on the effect of organic monomer/polymer additions on the properties of cement products have attracted the interest of researchers **(Singh ,2003)**. Cracks in concrete permit easier access of chloride ions, moisture, and oxygen to the reinforcing steel. Therefore, an effective corrosion- inhibiting admixture would need to remain effective when concrete is cracked **(Civjan ,2003)**.The corrosion behaviour of reinforcing steel embedded in concrete containing commercial corrosion inhibitor has received considerable attention in recent years**(kzair, 2001, Al–Hubboubi , 2001 , Amin ,2001 , Al-Ta'ie,2005,Brown,1999)**. However, very limited amount of work, have been carried out to investigate organic monomer as a corrosion inhibitor for reinforced steel in concrete **(Al-Hubboubi, 2002)**. This work, reports the effect of furfural on the fresh and hardened properties of concrete as well as its corrosion inhibition characteristics.

2. Experimental

2.1. Materials and proportion

Ordinary Portland cement Type (I) (Turabet-AL-Sabe'a) produced by Lebanon cement factory was used for concrete mixtures.Its chemical composition and physical properties are given in Tables 1 and 2 respectively.Natural sand of zone 3 and Crushed river gravel of 19mm maximum size conforming to Iraqi specification No.45/1980(IQS was used.A high range water reducing admixture (HRWR) known commercially as Eucobet super vz was used .Two reference mixes having characteristic compressive strengths of 35 and 45 MPa (A and B) have been prepared . Slump was kept within the range 60±15 mm.Commercial furfural was added to A and B mixes at different dosages (1, 2 and 3%) with and without HRWR.The details of mix proportions are given in Table3 .The pH of furfural

was found acidic (less than 5). It was neutralized by addition of 2.5% Na_2CO_3 to achieve a pH of around 7.

All specimens were wet-cured by covering the finished surface and molds with wet burlap. After one day, the specimens were de-molded and covered with wet burlap for two weeks and then left for air-curing under laboratory conditions for about two weeks, then the specimens were partially submerged in 3.5% NaCl solution for 350 days.

3. Experimental tests

Cubes with dimensions of 100*100*100 mm were prepared for the determination of compressive strength according to BS 1881 part 116 and initial surface absorption according to B.S 1881.part 5:1970. In order to identify the change in mineralogical composition upon furfural addition, x-ray diffraction analysis (XRD) was used. Two specimens, a reference of neat hydrated cement, and another with 3% added furfural were tested at 90-days age. The test was performed using a Chemadzu X-ray diffractometer-6000- Japan. Several electrochemical techniques were used for monitoring corrosion of steel in concrete such as half-cell potential, linear polarization techniques and the Tafel plot technique. The half-cell potential of the reinforcing steel was measured relative to copper-copper sulfate electrode (CSE) in accordance with the ASTM C876-99 standard. The corrosion rate measurements were conducted on specimens using a computerized potentiostat type Mlab200 of Bank Eleketronic, Germany. The test was conducted at 180 days of exposure to 3.5% NaCl saline solution. The reference electrode was saturated calomel (SCE); the auxiliary electrode was platinum electrode while the working electrode was the bar embedded in the concrete specimen. A good contact between the reference electrode and the concrete must be ensured in order to minimize the ohmic drops and avoid errors.The device was programmed to polarize the specimen potential to about 200 mV vs open circuit potential (OCP) in both the directions, i.e., cathodic and anodic. The time chosen scan was 10 mV /6.5 sec. The exposure area, (A) of steel embedded in concrete was calculated (1.25cm*3.14*18cm =70.65 cm^2), the corrosion current was divided on this area to obtain the corrosion current density. The Tafel curve and corrosion current were plotted and determined by the device. Polarization resistance was determined in the range± 30mV from the OPC.Current was linearly plotted against the potential. The polarization resistance Rp ($\Delta E/\Delta I$) is obtained from the slope of the polarization current curve near Ecorr in the cathodic and anodic direction. Tafel constants (anodic Tafel constant Ba and cathodic Tafel constant Bc) could be determined from the slopes of the straight line portions of a polarization curve. The polarization resistance Rp is related to the corrosion current Icorr through Stern-Geary relationship eq.(1).

$$\mathbf{Icorr} = \frac{B}{Rp} \quad \text{.............} \quad (1)$$

Where, B is the Stern–Geary constant.

$$\mathbf{B} = \frac{Ba * Bc}{2.3\,(Ba + Bc)} \quad \text{...........} \quad (2)$$

The corrosion current density, icorr is determined by dividing Icorr on the surface area, A. The corrosion rate in µm per year (µm/yr) is determined by multiplying icorr by 11.59 using Faraday's Law **(Trejo,2009)**.

4. Results and discussion

4.1 X-ray diffraction

The results of x-ray diffraction analysis (XRD) are shown in Fig.2.It is obvious that there exists some change in relative intensities of crystalline phases upon furfural addition. Moreover, a new crystalline (peak) phase has been detected. The change in peak intensity would be attributed either to a change in degree of crystallization of the specified phase and/or a change in its quantity. The relative intensity of $Ca(OH)_2$ at 2θ =34.11° (d=2.628 A) was reduced from 138 to 105 counts upon 3% furfural addition, while that of tobermorite phase ($C_5S_6H_5$) at 2θ =29.39° (d=3.03A) was increased from 124 to 160 counts.

2.3 Method of exposure

4.2 Water reduction due to furfural addition

The effect of furfural dosage on water reduction for concrete mixes with and without HRWR are shown in Fig.3 Results indicated that there would be a progressive water reduction as the dosage of furfural is increased. According to **(Singh ,2003)** the addition of water-soluble polymers permits the formation of highly workable paste with very little water. It seems that the additive acts as a lubricant. The water reduction was increased more by combined effect of furfural and HRWR than the effect of each admixture alone. The binary

admixture containing superplasticizer and polymer may result in certain beneficial effects to concrete that cannot be derived by using them individually **(Beaudoin,1989).**

4.3 Compressive strength

Compressive strength results for concrete mixes of 28 and 90 days are illustrated in Fig 4. These results indicate an increase in compressive strength upon furfural dosage increase. More improvement was observed due to the combined presence of furfural and HRWR .For 90 days results ,the compressive strength of concrete containing 1%HRWR and furfural was improved by a rate higher than HRWR -concrete compared to their mixes at 28 days.

4.4 Initial surface absorption (ISA)

The rate of water absorption by the surface zone of concrete under a fixed hydrostatic head is determined during a prescribed period between 10 minutes and two hours . The results indicated a decrease in ISA upon furfural dosage increase, Figs.(5 and 6).The percentage reduction in the ISA of 2 and 3% furfural-concrete (A2F and A3F) and for 2 hours duration of the test was 46.4 and 72.5% relative to its reference concrete (2A).This behavior is mainly attributed to the build-up of better microstructure, formation of additional C-S-H gel system and the filling of pores .The results had also shown that the percentage reduction in the ISA of HRWR-concrete (A1S) for 2 hours duration of the test , relative to its reference concrete was 49.3 % Further reduction in ISA was observed for A1S1F concrete compared to their reference concrete as well as to HRWR-concrete .The percentage reduction in 2 hours ISA of 2A1S1F was 70% compared to their reference . It is clear from these data that the synergistic effect of furfural and HRWR has a significant influence on the value of ISA of concrete.

4.5 Electrochemical measurements

4.5.1 Half-cell potential

The results of half-cell potentials of steel as referenced to a copper- copper sulphate electrode (CSE) are shown in Figs7 through 10. It is clear that the potentials of steel embedded in most of concretes were shifted towards negative potential at the beginning of exposure period. However, after ~30 days of exposure, the CSE potentials began to shift to more passive state. As per ASTM C876, -350 mV vs.CSE has been recognized as a threshold potential to reach an active condition of rebar .This level has been marked in the figures. It has been found that the addition of furfural improved the corrosion resistance of concretes, especially those with lower cement contents and weaker strengths. For example , in 35 MPa concrete (mix A) Fig7, the rebar embedded in concrete containing 2 and 3% furfural and reference concrete showed an initial potential values of -167,-197and -170 mV respectively. Specimens incorporating furfural, showed lower steel potential compared to reference concrete. However, rebar in reference concrete would tend to shift to more negative potential after about 150 days of exposure indicating a change in electrochemical status and the onset of the breakdown of passivity. At the end of 350 days of exposure the average potential of rebars in reference concrete (with cement content of 350 kg/m^3 and w/c of 0.563) reached -352.2 mV, while the average potentials of rebars embedded in 2 and 3% furfural –concrete reached -45 and -77 mV ,respectively. In 45 MPa concrete, Fig.8 , the higher cement content of reference concrete (with 450Kg/m^3 and w/c of 0.438) would initially provide a good protection to steel embedded in it, due to high alkalinity and denser structure that minimized chloride penetration towards the bars . However, the addition of 3% furfural (mix B3F) provided better protection and the best results were achieved by using 0.5% HRWR and 2% furfural (BS2F). The average potentials of bars in B, B3F, and BS2F at the end of 350 days were -72.5,-44 and +4 mV respectively. According to **(Babaei ,1986)**, positive half-cell potentials of small magnitude (generally less than +100mV) may result from large resistance in circuit (dry concrete or the absence of electrical continuity) or the absence of corrosion activity. It is suggested that such high reactivity contributes to the build-up of a protective passive layer. Hence, the trend of potentials of furfural-containing concrete shows some healing upon longer exposure duration. However, for furfural-free concrete, this layer does not exist, and the natural layer present due to high alkalinity of cement would be destroyed upon longer exposure duration. Accordingly, the potentials drop down to the level of high probability of corrosion. Reinforcing steel in HRWR- concrete exhibited lower potentials than those of steel embedded in reference concretes (mixes A and B).This behavior could be related to the reduction in water content and the formation of the denser gel. However , bars in HRWR- concrete of 35MPa were shifted to a more negative potential after about 280 days of exposure, refer to Fig.9.According to (Neville,2005) superplasticizer per se does not

affect the pore structure and, therefore, does not alter the process of corrosion. The results also demonstrated that the combined action of furfural and HRWR provide excellent protection to steel. The average potentials of bars in A1S , A1S1F, A1S2F and A1S3F at the end of 350 days were -222.5 , -55.5, -62 and - 62.5 mV, respectively .However, the efficiency of combined effect of furfural and HRWR in concrete containing higher cement content (450 kg/m^3) was affected by furfural dosages .The B1S3F mix provided little protection to rebar at the beginning of exposure as an evidence of concrete weakness but with time it became stronger and exhibited an excellent protection to the rebar, Fig.10 .This behavior may be attributed to the extent of polymerization of furfural with time.

4.5.2 Polarization measurement

4.5.2-1 Tafel Plots

The Tafel plots for reinforced concrete specimens were tested at age 180days of partial submergence in to 3.5%NaCl solution. Tafel plots for specimens represent the relation between the current (µA) and the potential (-mV) in a semi-logarithm scale. They are shown in Figs11through14. These figures were used to obtain the Tafel plots parameters: Ba, Bc, B and i_{corr} which represent respectively the anodic Tafel slope, cathodic Tafel slope, the B constant and the corrosion current. These parameters are listed in Table 4 as well as the current density (µA/cm^2) which can be obtained by dividing i_{corr} in (µA) by exposed surface area of the embedded reinforcing steel in concrete (cm^2).The results confirm that the corrosion process of the steel reinforcement for all specimens were anodically controlled depending on the Tafel slopes with the conclusion that the anodic process is the slower process (the one with the large polarization), whereas the rate of oxygen diffusion through the concrete determines the rate of corrosion. The constant B is dependent on the magnitudes of the Tafel slopes of the anodic and cathodic reactions characterizing the particular corroding system.

4.5.2.2. Linearpolarization resistance measurement

The linear polarization resistance represents the electrical resistivity of the passive film at the surface of the re-bars, and is determined graphically from the slope of the potential (mV)-current density (µA/cm^2) curves. According to **(Mancio,2005)** the greater the slope, the more difficult for the charge to transfer across the metal/electrolyte interface, and therefore the corrosion current density is smaller and, consequently, the corrosion reaction rate is slower. The potential –current (E-I) relationships for 35 MPa concrete and 45MPa concretes with and without furfural ,HRWR are given in Table 4 and shown in Figs.15 and 16 . Results showed that polarization resistance for 45 MPa - concrete (2.069 KΩ) was higher than 35MPa- concrete (1.685 KΩ).This improvement is due to higher cement content and lower w/c ratio. Polarization resistance measurements showed that furfural-treated concrete specimens had higher Rp values compared with the reference concrete. An additional improvement was achieved by combined effect of furfural and HRWR over HRWR-concrete. The calculated corrosion density determined by using Stern-Geary equation for reinforcement steel of 35 and 45 MPa-concretes showed that with an increase in cement content and a decrease in w/c ratio ,a decrease in corrosion current density was achieved,Fig.17. This improvement results from the development of passivity due to high alkalinity and dense structure. The use of 2 and 3% furfural resulted in 36.4 and 63% reduction in corrosion current density of steel in A2F and A3F mixes respectively compared to A(35 MPa) concrete. Similarly, the addition of 3% furfural to 45 MPa concrete led to a reduction in corrosion current density of about 64%.Less effectiveness was observed by the combined effect of furfural and HRWR .However a significant reduction in corrosion current density was achieved by 2%furfural and HRWR in B1S2F over HRWR-concrete B1S.

5. Conclusions

1- A progressive water reduction would take place as the dosage of furfural is increased. The water reduction was increased more by combined effect of furfural and HRWR than the effect of each admixture alone.

2- A decrease in ISA upon furfural dosage increase was observed .The percentage reduction in the ISA of 2 and 3% furfural-concrete (A2F and A3F) and for 2 hours duration of the test was 46.4 and 72.5% relative to its reference concrete (A).This behavior is mainly attributed to the build-up of better microstructure, formation of additional C-S-H gel system and the filling of pores .The results had also shown that the percentage reduction in the ISA of HRWR-concrete (A1S) for 2 hours duration of the test , relative to its reference concrete was 49.3 % Further reduction in ISA was observed for A1S1F concrete compared to

their reference concrete as well as to HRWR-concrete .The percentage reduction in 2 hours ISA of 2A1S1F was 70% compared to their reference . It is clear from these data that the synergistic effect of furfural and HRWR has a significant influence on the value of ISA of concrete.

3 Reinforcing steel in HRWR- concrete exhibited lower potentials than those embedded in reference concretes (35 and 45 MPa). However, rebars in HRWR- concrete of 35MPa were shifted to a more negative potential after about 280 days of continuous exposure to 3.5% NaCl solution.

4-The combined presence of furfural and HRWR was found to provide an excellent corrosion protection to steel. The potentials of rebar in A1S, A1S1F, A1S2F and A1S3F-concretes at the end of 350 days were -222.5,-55.5,-62 and -62.5 mV, respectively.

5-After six-month period of continuous exposure to 3.5% NaCl solution, electrochemical measurements confirmed that the utilization of furfural in concrete would result in a significant reduction in corrosion tendency. The effective dosage was 3% by weight of cement. Furfural dramatically reduces the rate of corrosion; the inhibition efficiencies were 62.7 and 63.8 % due to 3% furfural addition to 35- and 45-MPa-concretes respectively.

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Table 1: Chemical composition and main compounds of cement.

Oxides composition ,%	Turabet Al-Sabe'a OPC	Limits of (IQS) No.5/1984
CaO	63.31	-
SiO_2	19.44	-
Al_2O_3	5.12	-
Fe_2O_3	3.57	-
MgO	1.07	$\leq$5.00
SO_3	2.8	$\leq$2.80
L.O.I.	3.87	$\leq$4.00
Lime Saturation Factor,L. S. F.	0.975	0.66-1.02
C_3S	62.44	-
C_2S	8.97	-
C_3A	7.53	-
C_4AF	10.85	-

Table 2: Physical properties of cement

Physical Properties	Turabet Al-Sabe›a OPC	Limits of (IQS) No.5/1984
Specific surface area (Blaine method), cm^2/gm	3203	$\geq$2300
Setting time(Vicate apparatus), Initial setting, hour: minute Final setting, hour: minute	 2:05 3:50	 $\geq$00:45 $\leq$10:00
Compressive strength ,MPa 3 days 7 days	 28.6 30	 $\geq$15 $\geq$23

Table3: Concrete mixes

Symbol	Mix components						w/c	Slump (mm)
	Cement kg/m³	Sand kg/m³	Gravel kg/m³	Water kg/m³	Furfural % of cement Wt.	HRWR % of cement Wt.		
A	350	845	1033	197	-	-	.563	50
A2F	=	=	=	189.6	2	-	.542	65
A3F	=	=	=	182.3	3	-	.521	60
A1S	=	=	=	171.3	-	1	.49	80
A1S1F	=	=	=	162.6	1	1	.465	60
A1S2F	=	=	=	157.5	2	1	.45	55
A1S3F	=	=	=	156.1	3	1	.446	50
B	450	741.5	1033	197	-	-	.438	55
B3F	=	=	=	189.5	3	-	.421	55
BS2F	=	=	=	177.3	2	.5	.394	60
B1S	=	=	=	171	-	1	.38	50
B1S1F	=	=	=	197	1	1	.36	50
B1S2F	=	=	=	189.5	2		.35	75
B1S3F	=	=	=	177.3	3	1	.37	60

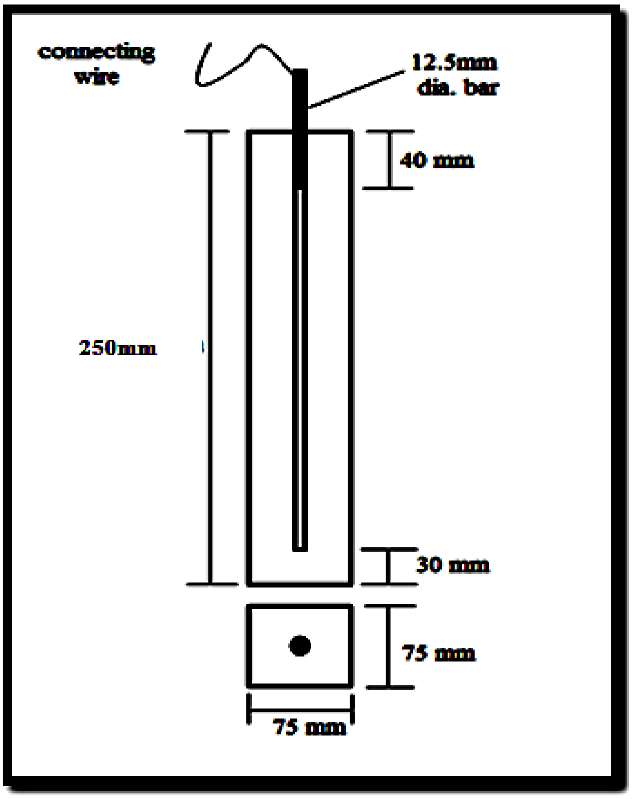

Fig.1: Sketch of reinforced concrete prism

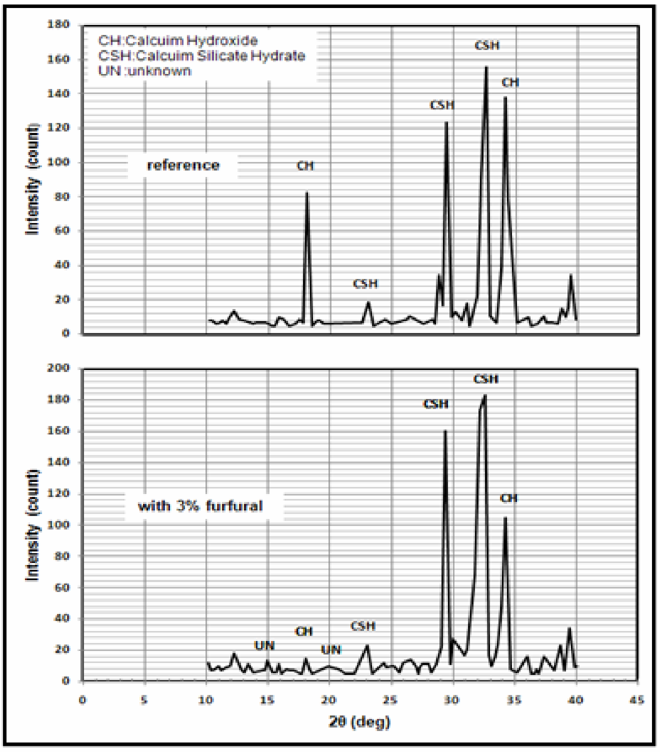

Fig.2: X-ray diffraction of neat hydrated cement with and without furfural.

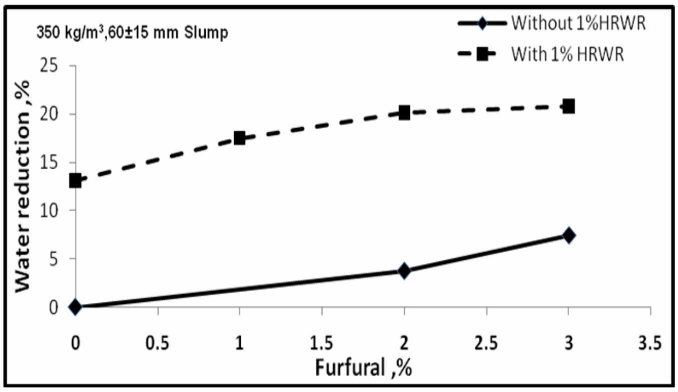

Fig.3: Effect of furfural dosage and combined effect of furfural and 1% HRWR on water reduction

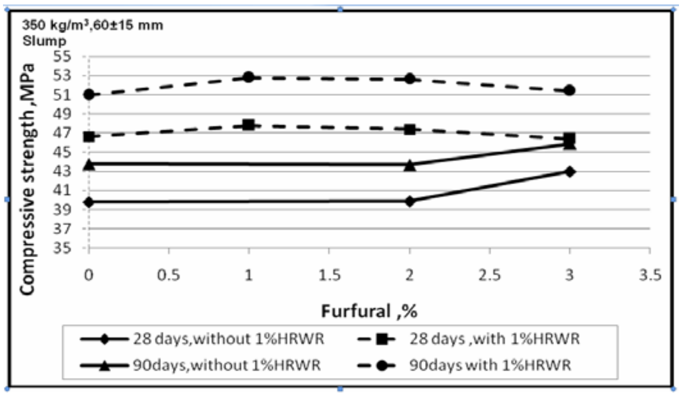

Fig. 4: Effect of furfural dosage on compressive strength of 1% HRWR –containing concretes.

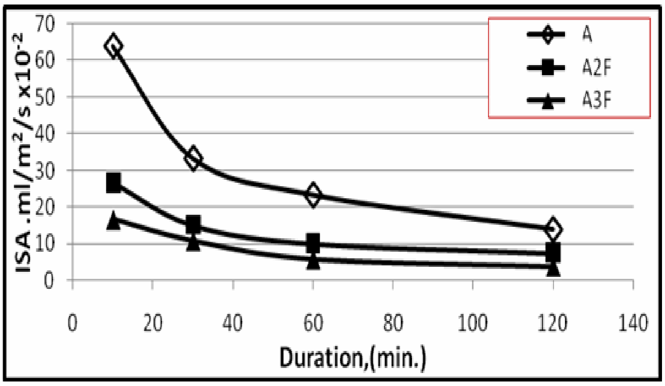

Fig.5 : Effect of furfural on initial surface absorption values at age of 28days

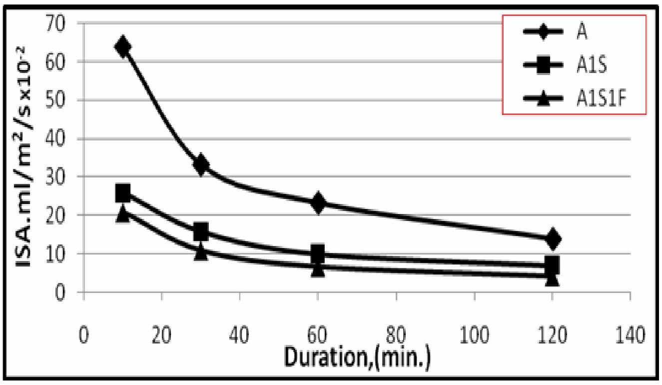

Fig.6: Combined effect of furfural and HRWR on initial surface absorption at age of 28days

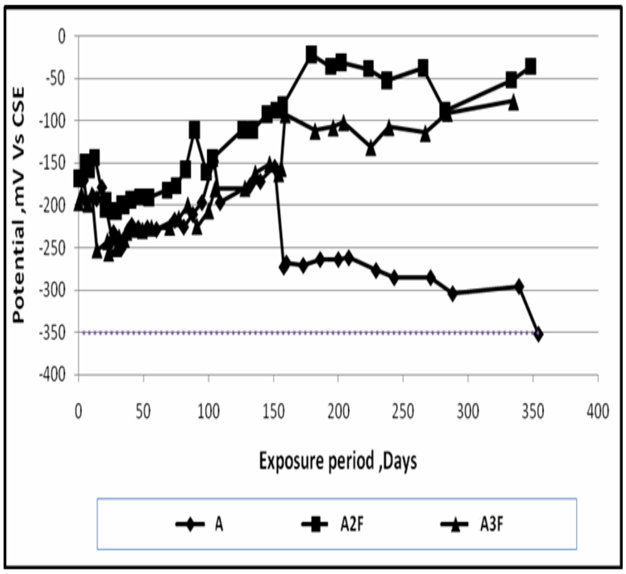

Fig.7: Potential versus time behavior of rebar embedded in furfural-concrete of 35MPa

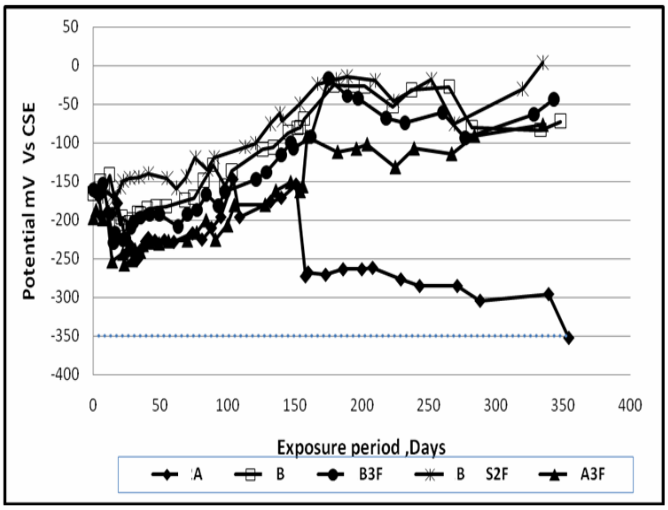

Fig.8: Potential versus time behavior of rebar embedded in furfural-concretes of 35MPa and 45 MPa.

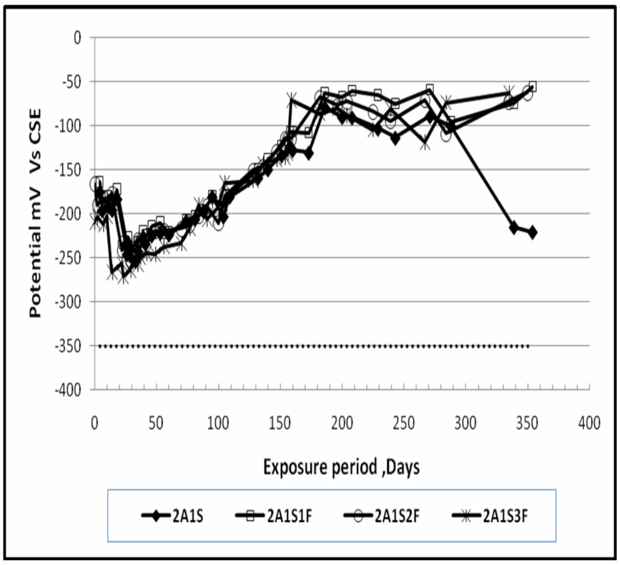

Fig.9: Potential versus time behavior of rebar embedded in HRWR and furfural-concrete of 35M

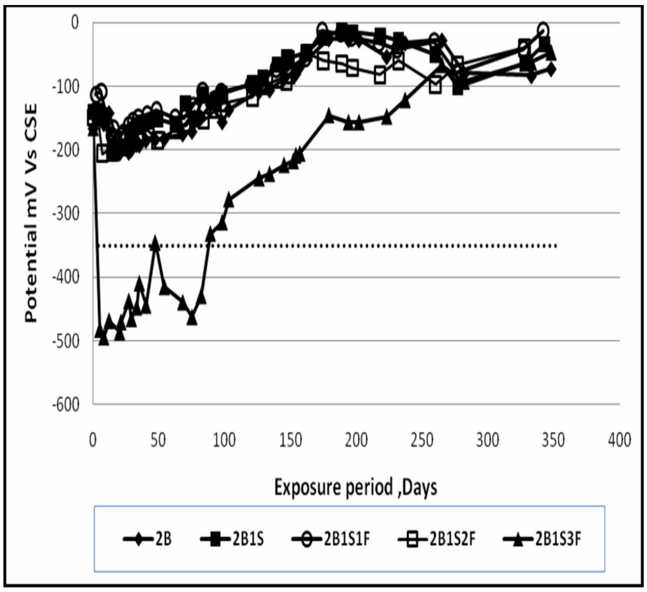

Fig.10: Potential versus time behavior of rebar embedded in HRWR and furfural-concrete of

Table 4:Tafel constant and linear polarization resistance results

Symbol	Tafel plot						Linear polarization resistance			
	E_{corr} SCE -mV	Tafel constant mV/decade			Corrosion current I_{corr} µA	Corrosion current density µA/cm²	Polarization resistance Rp-KΩ	Corrosion Current I_{corr} µA	Corrosion current density i_{corr} µA/cm²	Corrosion rate µm/yr
		Ba	Bc	B				B/Rp		
A	360	300	130	39.4	19	.27	1.685	23.4	.33	3.83
A2F	270	275	90	29.5	10	.14	2.183	14.8	.21	2.44
A3F	300	400	70	25.9	8	.11	2.970	8.7	.123	1.43
A1S	300	370	85	30.1	9	.13	2.609	11.5	.163	1.9
A1S2F	250	450	100	35.6	9	.13	3.158	11.3	.16	1.86
A1S3F	260	450	110	38.4	9	.13	3.158	12.2	.173	2
B	295	330	90	30.7	10.2	.144	2.069	14.8	.21	2.44
B3F	300	190	90	26.6	4	.057	4.918	5.4	.076	.88
B1S	240	250	115	34.2	7.5	.106	3.186	10.8	.153	1.77
B1S1F	200	255	115	34.5	6.5	.092	3.297	10.4	.147	1.7
B1S2F	300	230	110	32.3	4	.057	5.000	6.46	.09	1.044

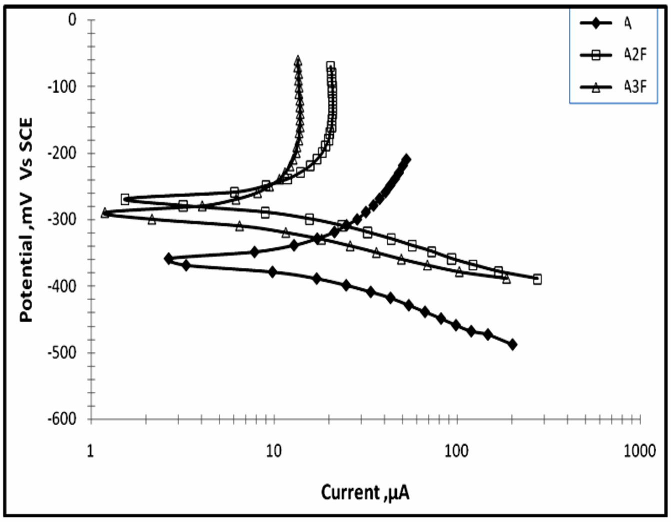

Fig.11: Tafel plot for steel in furfural – concrete (350 kg/m3)]at 180 days of partially submergence in 3.5%NaCl solution.

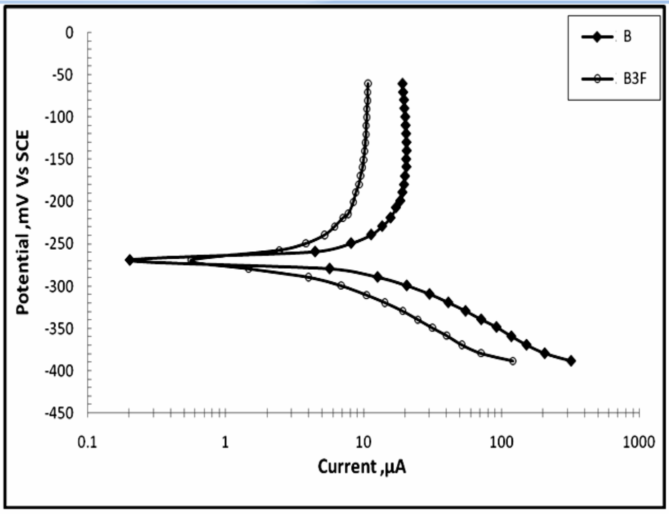

Fig.12:Tafel plot for steel in furfural –concrete(450 kg/m3) at 180 days of partially submergence in 3.5%NaCl solution.

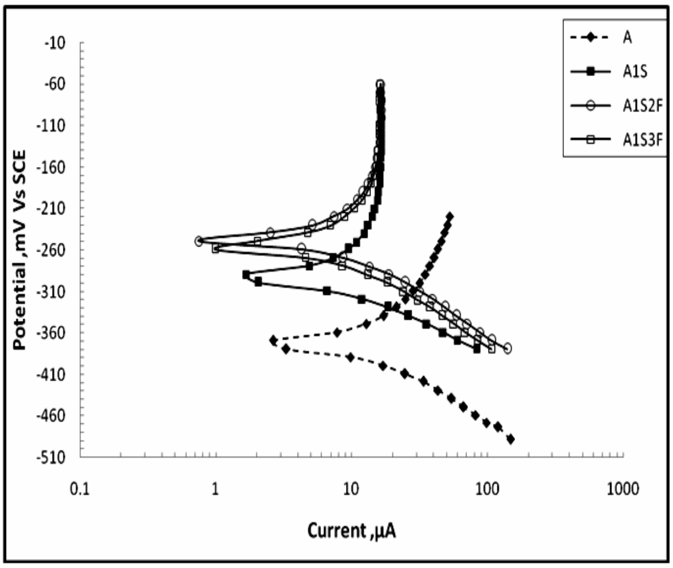

Fig.13: Tafel plot for steel in furfural – concrete (350 Kg/m3) with and without HRWR at 180 days of partially submergence in 3.5%NaCl solution.

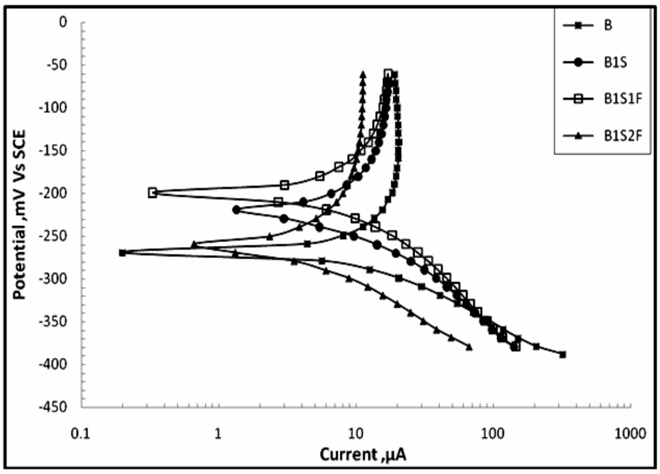

Fig.14:Tafel plot for steel in furfural-concrete (450 Kg/m3)with and without HRWR at 180 days of partially submergence in 3.5%NaCl solution.

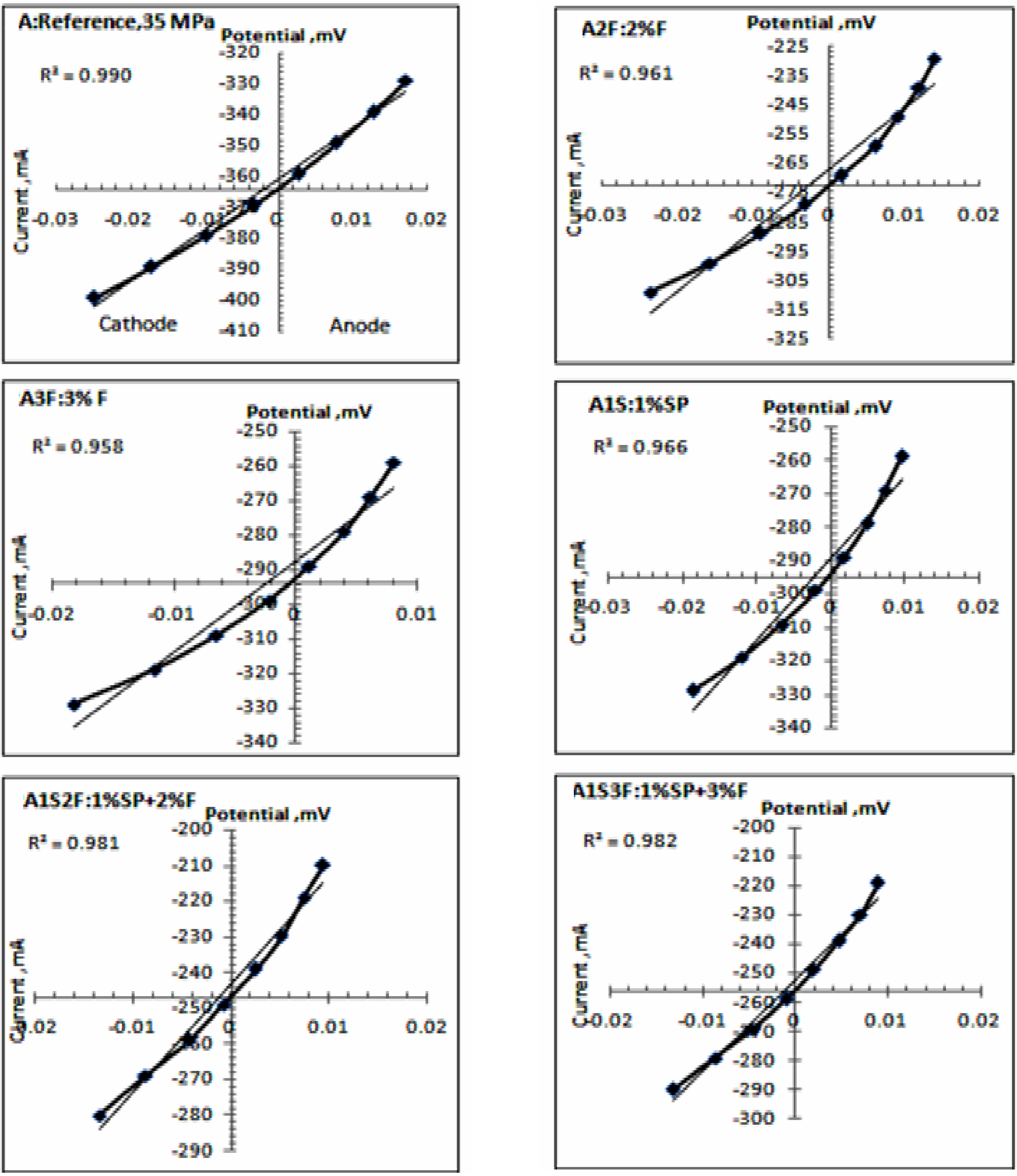

Fig.15: Polarization resistance of steel bars in 35 Mpa reference concrete and concretes with different admixtures.

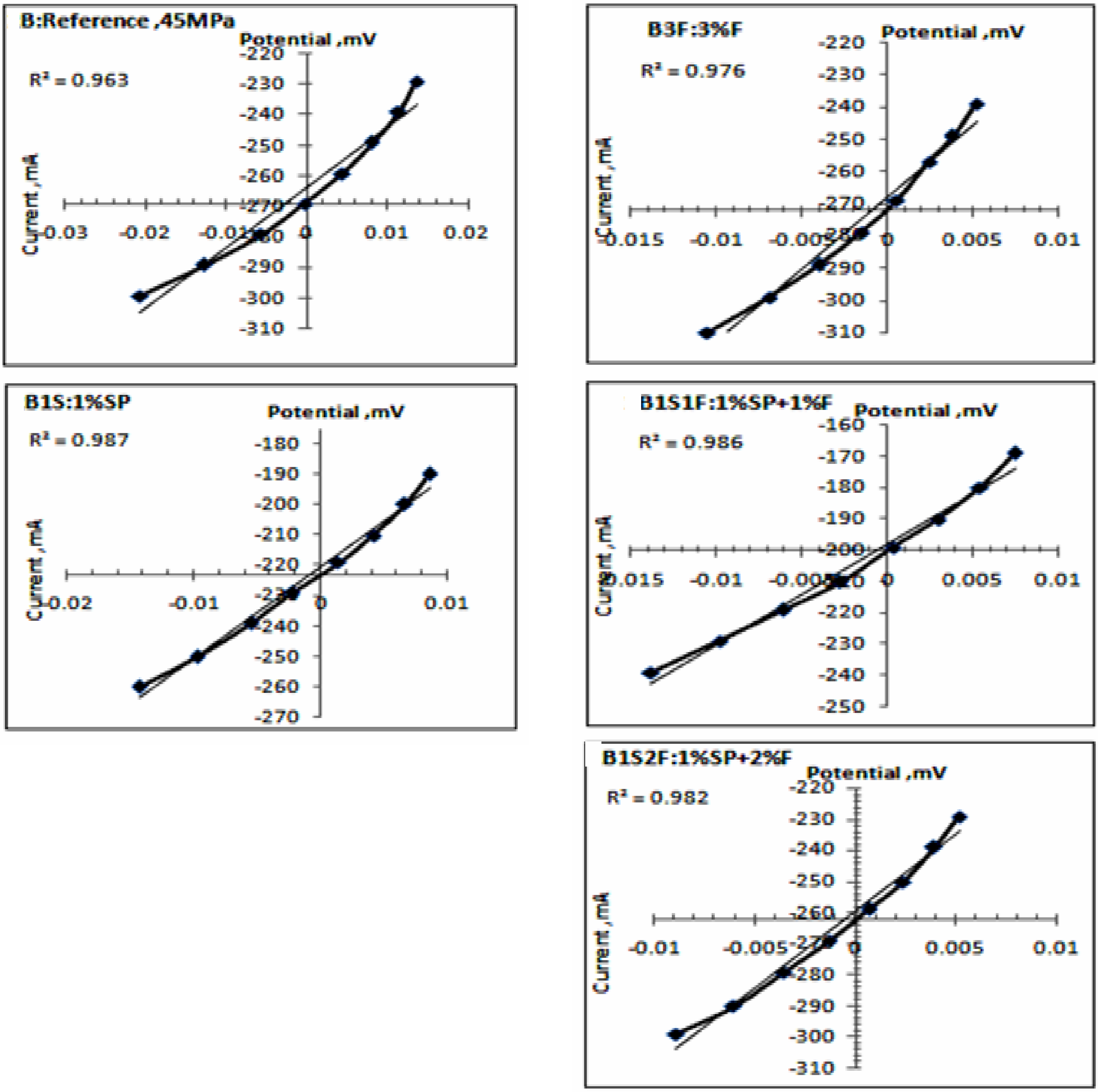

Fig. 16.Polarization resistance of steel bars in 45 MPa reference concrete and concretes with different admixtures...

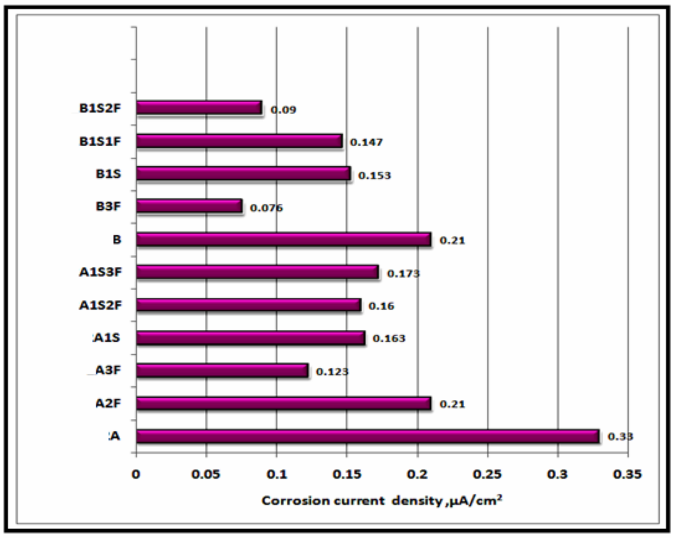

Fig.17: Corrosion current density as determined by using Stern-Geary equation (i_{corr}=B/Rp.A) for bars in tested concretes.