# Investigation of Corrosion Potential Probability and Concrete Resistivity of Inhibited Reinforcement Chloride threshold in Corrosive Environment

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#### **Abstract**

This study investigated the effects of chloride attack on reinforcing steel embedded in reinforced concrete structures built in the marine environment. An experimental work simulated the quick process by acceleration process on non-inhibited and inhibited reinforcement of acardium occidentale l. resins extracts with polished thicknesses of 150µm, 250µm and 350µm, embedded in concrete slab and immersed in sodium chloride (NaCl) and accelerated for 119 days using Wenner four probes method; it was done by placing the four probes in contact with the concrete directly above the reinforcing steel bar and assessed the actions of half cell potential, concrete resistivity and tensile strength of reinforcement to corrosion. Results recorded of half cell potential, concrete resistivity and tensile strength properties for non- inhibited concrete specimens on the mapping areas for the expedited periods designated 95% probability of corrosion and indicating a high or moderate probability of corrosion. Results recorded of potential  $E_{corp}^{mV}$ , concrete resistivity and tensile strength of Acardium occidentale l. inhibited specimen, indicated a 10% or uncertain probability of corrosion which indicates no corrosion presence or likelihood and concrete resistivity indicated a low probability of corrosion or no corrosion indication. Average percentile results of potential E<sub>corr,mV</sub>, and concrete resistivity are 27.45% and 68.45% respectively. When compared to corroded samples, corroded has 75.4% increased values potential  $E_{corr,mV}$  and 33.54% decreased values of concrete resistivity, yield stress against ultimate strength at in comparison to corrode as 100% nominal yield stress decremented from 108.38% to 90.25% respectively, weight loss at 69.3% against 43.98% and 51.45% to 89.25%, cross-sectional diameter reduction, both showed decreased values of corroded compared to coated specimens.

Key Words: Corrosion, Corrosion inhibitors, corrosion potential, concrete resistivity and Steel Reinforcement.

# 1.0 Introduction

Chloride causes damage to reinforcing steel by attacking the passive film and corrosion starts at certain locations along the steel bar where the loss in passive layer occurs. If the level of chlorides is high compared with available hydroxyl ions the affected area becomes more acidic and chloride rich leading to further breakdown in the passive layer and a decrease in corrosion potential with increasing anodic activity in the form of iron (Thangavel and Rengaswamy [1], Ann and Song [2]). In addition, hydroxyl ions are formed due to cathodic reactions leading to increase in the cathodic area. The small anode to cathode area further promotes corrosion, which result in an increase in the corrosion current density Frazeck [3]. These processes encourage the formation of corrosion pits and substantial local loss of cross-sectional area of steel bar, and hence increase aggravation of the corrosion process. This, however, can be reversed by the available hydroxyl ions that have the ability to neutralize the acidity, stabilize and repair the damaged parts of the passive film through further iron oxide deposition. The risk of corrosion increases as the chloride content increases and when the chloride content at the surface of the steel exceeds a certain limit, called the threshold value, corrosion will occur if the water and oxygen are also available (Thomas [4], Glass and Buenfeld [5], Ann and Song [2]).

There have been numerous studies undertaken to determine the effect of chloride concentration on corrosion of reinforcing steel in alkaline solutions, with the purpose to establish unique critical chloride for pitting initiation (Li and Sagues [6], Saremi and Mahallati [7]). However, the chloride threshold depends on several variables and, for this reasons values reported by different researchers showed a significant sector. It was reported Li and

Sagues [6], that the critical concentration of chloride in simulated concrete pore solutions with pH = 13.6 would be 0.4 to 0.6M (mole/liter), while in saturated calcium hydroxide  $Ca(OH)_2$  somewhat between 0.01 to 0.04M. It has been also found (Saremi and Mahallati [7]) that the breakdown of passive film on mild steel in  $Ca(OH)_2$  is at [CU]/[OH] ratio of 0.60. Hausman [8] reported that steel immersed in a pH =13.2 solution with the addition of 0.25M NaCl remained in the passive state while Goudi (1970) found that the maximum amount of sodium chloride that can be tolerated in a NaOH solution with pH =13.9 was 0.12M. Moreno *et al.* [9] reported that the existence of a passivity breakdown potential due to pitting corrosion for as-received steel immersed in saturated  $Ca(OH)_2$  containing 0.05% chloride concentration.

Aprael and Hasan [10] performed anodic polarization tests on mild steel in saturated  $Ca(OH)_2$  solution with 0.10 to 3% NaCl by weight of water, and concluded that a thin passive film of few nano meters (nm) of oxides covers the metal surface. This layer is responsible for the passive nature of the metal at low level of NaCl, and increasing NaCl content to 3% NaCl destroys the passive film and shifts the corrosion potential to more negative value of  $\sim$  550mV. The rate of oxide layer destruction rises with increase the exposure time of specimen to chloride solution and subsequently decreases in corrosion potential and increases the corrosion rate Abd ELhaleem *et al.* [11].

# 2.0 MATERIALS AND METHODS FOR EXPERINMENT

# 2.1 Aggregates

The fine aggregate was gotten from the river, washed sand deposit, coarse aggregate was granite a crushed rock of 12 mm size and of high quality. Both aggregates met the requirements of [12]

#### **2.1.2 Cement**

The cement used was Ordinary Portland Cement, it was used for all concrete mixes in this investigation. The cement met the requirements of [13]

#### **2.1.3 Water**

The water samples were clean and free from impurities. The fresh water used was gotten from the tap at the Civil Engineering Department Laboratory, University of Uyo, Uyo. Akwa - Ibom State. The water met the requirements of [14]

#### 2.1.4 Structural Steel Reinforcement

The reinforcements are gotten directly from the market in Port Harcourt. [15]

## 2.1.5 Corrosion Inhibitors (Resins / Exudates) Acardium occidentale 1.

The study inhibitor Acardium occidentale 1.is of natural tree resin /exudate substance extracts. They are abundantly found in Rivers State bushes and they are sourced from plantations and bushes of Odioku communities, Ahoada West Local Government areas, Rivers State, from existed and previously formed and by tapping processes for newer ones.

# 2.2 EXPERIMENTAL PROCEDURES

# 2.2.1 Experimental method

# 2.2.2 Sample preparation for reinforcement with coated resin/exudates

Corrosion test was conducted on high tensile reinforcing steel bar of 12mm, specimens rough surface were treated with sandpaper and wire brush, washed with acetone to remove rust and dried to enable proper adhesion of coated / inhibitive materials. Coating was done by direct application on the ribbed reinforcement rough surface with 150µm, 250µm and 350µm coated thicknesses of Acardium occidentale 1. paste were polished and allowed to dry for 72 hours before embedded into concrete slab.

Mix ratio of 1:2:3 by weight of concrete, water cement ratio of 0.65, and manual mixing was adopted. The samples were designed with sets of reinforced concrete slab of 150mm thick x 350mm width x 900mm long, uncoated and coated specimens of above thicknesses were embedded into the concrete, spaced at 150mm apart. Fresh concrete mix batch were fully

compacted to remove trapped air, with concrete cover of 15mm and projection of 150mm for half cell potential measurement and concrete resistivity tests. Slabs were demoulded after 72 hours and cured for 28 days with room temperature and corrosion acceleration ponding process with Sodium Chloride lasted for 119 days with 14 days checked intervals for readings. The corrosion rates were quantified predicated on current density obtained from the polarization curve and the corrosion rate quantification set-up. The corrosion cell consisted of a saturated calomel reference electrode (SCE), counter electrode (graphite rod) and the reinforcing steel embedded in concrete specimen acted as the working electrode. The polarization test was performed utilizing scanning potential of -200 mV through 1200mV, with a scan rate of 1mV/s. The data were recorded for a fine-tuned duration of 1hr at ambient temperature. The polarization curve was obtained as the relationship between corrosion potential and current density.

## 2.3 Accelerated Corrosion Test

In order to test concrete resistivity and durability against corrosion, it was necessary to design an experiment that would accelerate the corrosion process and maximize the concrete's resistance against corrosion until failure. The accelerated corrosion test allows the acceleration of corrosion to reinforcing steel embedded in concrete and can simulate corrosion growth that would occur over decades. A laboratory acceleration process helps to distinguish the roles of individual factors that could affect chloride induced corrosion. An accelerated corrosion test is the impressed current technique which is an effective technique to investigate the corrosion process of steel in concrete and to assess the damage on the concrete cover. (Care and Raharinaivo [16] Reinforcement corrosion normally requires long exposure period of time, and usually by the first crack observed on the concrete surface. Therefore, for design of structural members and durability against corrosion as well as selection of suitable material and appropriate protective systems, it is useful to perform accelerated corrosion tests for obtaining quantitative and qualitative information on corrosion resistance in a relatively shorter period of time.

## 2.4 Corrosion Current Measurements (Half-cell potential measurements)

Half-cell potential measurements are indirect method of assessing potential bar corrosion, but there has been much recent interest in developing a means of performing perturbative electrochemical measurements on the steel itself to obtain a direct evaluation of the corrosion rate (Gowers and Millard [17]). Corrosion rates have been related to electrochemical measurements based on data first reported by Stern and Geary [18]. If the potential measurements indicate that there is a high probability of active corrosion, concrete resistivity measurement can be subsequently used to estimate the rate of corrosion. This was also stated from practical experience (Figg and Marsden [19] and Langford and Broomfield [20]. Classifications of the severity of rebar corrosion rates are presented in Table 2.1. However, caution needs to be exercised in using data of this nature, since constant corrosion rates with time are assumed.

Table 2.1: Dependence between potential and corrosion probability

Potential $E_{\rm corr}$	Probability of corrosion
Ecorr < -350mV	Greater than 90% probability that reinforcing steel corrosion is occurring in that area at the time of measurement
$-350 \text{mV} \le E c_{\text{orr}} \le -200 \text{mV}$	Corrosion activity of the reinforcing steel in that area is uncertain
$E_{\rm corr} > -200 {\rm mV}$	90% probability that no reinforcing steel corrosion is occurring in that area at the time of measurement (10% risk of corrosion

## 2.5 Concrete Resistivity Measurement Test

In the study, the Wenner four probes method was used; it was done by placing the four probes in contact with the concrete directly above the reinforcing steel bar. Different readings were taken at different locations at the surface of the concrete. The mean values of the readings were

recorded as the final readings of the resistivity in the study. The saturation level of the slabs was monitored through concrete electrical resistivity measurements, which are directly related to the moisture content of concrete. The electrical resistivity becomes constant once the concrete has reached saturation. Before applying water on the slabs, the concrete electrical resistivity was measured in the dry condition at the specified locations. Henceforth, these measurements will be referred to as the measurements in «dry» conditions. These locations were chosen at the side of the slabs, since concrete electrical resistivity measurements could be taken when water was on the top surface of the slab. Time limitation was the main challenge to perform all the experimental measurements, as the concrete saturation condition changes with time. After applying water on the surface of the slabs, the concrete resistivity was measured daily at the reference locations, looking for the saturation condition. Since each of the slabs had a different w/c, the time needed to saturate each of the slabs was not the same. Once one slab would reach the saturated condition, the water could be drained from that slab, while the other slabs remained ponded.

Table 2.2: Dependence between concrete resistivity and corrosion probability

Concrete resistivity $ ho$ , k $\Omega$ cm	Probability of corrosion
ρ < 5	Very high
$5 < \rho < 10$	High
$10 < \rho < 20$	Low to moderate
$\rho > 20$	Low

## 2.6 Tensile Strength of Reinforcing Bars

To ascertain the yield and tensile strength of tension bars, bar specimens of 12 mm diameter of non-corroded, corroded and coated were tested in tension in a Universal Testing Machine and were subjected to direct tension until failure; the yield, maximum and failure loads being

recorded. To ensure consistency, the remaining cut pieces from the standard length of corroded and non-corroded steel bars were subsequently used for mechanical properties of steel.

# 3.0 Experimental results and discussion

The results of the half-cell potential measurements in table 3.1 were plotted against concrete resistivity of table 3.2 for easy interpretation. It is evident that potential  $E_{\rm corr}$  if low (< -350mV) in an area measuring indicates a 95% probability of corrosion. In the other measuring points, potential  $E_{\rm corr}$  is high (-350mV  $\leq E_{\rm corr} \leq$  -200mV), which indicates a 10% or uncertain probability of corrosion

Results of the concrete resistivity measurements are shown in Table 3.2. It used as indication of likelihood of significant corrosion ( $\rho < 5$ ,  $5 < \rho < 10$ ,  $10 < \rho < 20$ ,  $\rho > 20$ ) for Very high, High, Low to moderate and Low, for Probability of corrosion. Resistivity survey data gives an indication of whether the concrete condition is favorable for the easy movements of ions leading to more corrosion. Concrete resistivity is commonly measured by four-electrode method.

# 3.1 Non-corroded Concrete Slab Members

Results obtained from table 3.1 of half-cell potential measurements for and concrete resistivity for 7days to 119 days respectively indicated a 10% or uncertain probability of corrosion which indicates no corrosion presence or likelihood and concrete resistivity which indicated a low probability of corrosion or no corrosion indication.

Table 3.1, 3.2 and 3.2 are the results of derived from computed percentile average values of randomly cast specimens slab samples from A-I of control, corroded and coated specimens of 150μm, 250μm, 350μm forming A, B and C from ABC, DEF and GHI. Figures 3.1 – 3.6 are the plots of concrete resistivity  $\rho$ , kΩcm versus Potential  $E_{corr}^{mV}$  Relationship which showed average of 27.2% Potential  $E_{corr}^{mV}$  and 87.8% Concrete Resistivity. Figures 3.3 and 3.4 are the plots of yield stress versus ultimate strength of with values of 100% normal yield stress of 410N/mm<sup>2</sup> and 100.68%. Figures 3.5 and 3.6, the graphical presentation of weight loss against cross-section diameter reduction at 67.1% and 98.2% respectively.

## 3.2 Corroded Concrete Slab Members

Tables 3.1, 3.2 and 3.3 are the results recorded of potential  $E_{corr}$ ,  $^{mV}$ , and concrete resistivity and tensile strength properties for non-inhibited concrete specimens on the mapping areas for the

expedited periods of 7days to 119 days which designated 95% probability of corrosion and betokening a high or moderate probability of corrosion. Average results on comparison showed incremental values of 70.1% against 27.2% non-corroded of Potential  $E_{corr}$ ,  $^{mV}$  and 87.8% to 38.8%, decremented values in concrete resistivity. Figures 3.1 and 3.2 are the plots representations of concrete resistivity  $\rho$ ,  $k\Omega$ cm versus Potential  $E_{corr}$ ,  $^{mV}$  Relationship. Figures 3.3 and 3.4 are the plots of yield stress against ultimate vigor at summary and average state of corroded slab with nominal values of 100% and decremented in ultimate vigor from 100.68% to 96.12%, while figures 3.5 and 3.6 presented the weight loss versus cross-section diameter reduction decremented due to assail from sodium chloride from 67.1% to 48.5% and 98.2% to 94.82% respectively.

## 3.3 Acardium occidentale 1.Steel Bar Coated Concrete Cube Members

Tables 3.1, 3.2 and 3.3 are the results recorded of potential Ecorr,mV, and concrete resistivity and tensile strength of Acardium occidentale Linhibited specimen, the results indicated a 10% or uncertain probability of corrosion which indicates no corrosion presence or likelihood and concrete resistivity indicated a low probability of corrosion or no corrosion indication. Average percentile results of potential  $E_{corr,mV}$ , and concrete resistivity are 27.45% and 68.45% respectively. When compared to corroded samples, corroded has 75.4% increased values potential  $E_{corr,mV}$  and 33.54% decreased values of concrete resistivity . Figures 3.1 and 3.2 are the plots representations of Concrete Resistivity  $\rho$ , k $\Omega$ cm versus potential  $E_{corr,mV}$  Relationship. Figures 3.3 and 3.5 represented the plots for arbitrarily and computed percentile average values of yield stress against ultimate strength at in comparison to corrode as 100% nominal yield stress decremented from 108.38% to 90.25% and figures 3.5 and 3.6 respectively presented weight loss at 69.3% against 43.98% and 51.45% to 89.25%, cross-sectional diameter reduction, both showed decreased values of corroded compared to coated specimens.

Table 3.1: Potential  $E_{corr}$ , after 28b days curing and 115 days acceleration Ponding

s/no	Inhibitor (resin/exudates) and controlled sample		Potential $\mathbf{E}_{\mathrm{corr,mV}}$ Time Intervals after 28 days curing								
		A (7days)	B (21days)	C (35days)	D (49days)	E (63days)	F (77days)	G (91days)	H (105 days)	I (119 days)	
1	Control Concrete slab	-102	-102.2	-100.3	-101.2	-101.7	-100.8	-100.3	-101.4	-100.4	
2	Non-inhibitor	-268.5	-294.7	-328.6	-367.7	-377.5	-384.5	-418.4	-425.6	-429.7	
		150µm,			250µm,			`350µm,			
3	Acardium occidentale 1.	-104.5	-103.5	-104.2	-101.	-114.7	-103.7	-116.5	-100.8	-108.5	
			Ave	rage val	ues Potei	ntial E <sub>cor</sub>	r,mV		1		
			ABC = A			`DEF = B			GH1 = C		
1A	Control Concrete slab	-101.5			-102.2			-100.7			
2A	Non-inhibitor	-297.3			-393.5			-424.6			
		150µm,				250μm,			`350µm,		
3A	Acardium occidentale 1		-110.7			-111.4			`-116.8		

Table 3.2 : Results of Concrete Resistivity  $\rho,\,k\Omega cm$  Time Intervals after 28 days curing and 115 days acceleration ponding

s/no	Inhibitor (resin/exudates)	
	and controlled sample	Concrete Resistivity ρ, kΩcm
		Time Intervals after 28 days curing

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		A	В	С	D	Е	F	G	Н	Ι	
		(7days)	(21days)	( 35days)	( 49days)	(63days)	( 77days)	(91days)	(105 days)	(115 days)	
1	Control Concrete slab	15.35	15.52	15.42	15.65	15.48	14.43	15.45	15.45	15.48	
2	Non-inhibitor	6.77	6.91	7.74	8.05	8.22	8.38	9.12	9.55	9.59	
		150μm,			250µm,			`350μm,			
3	Acardium occidentale 1.	13.3	13.22	13.41	14.18	14.26	14.44	14.46	14.58	14.32	
	Average values Concrete Resistivity $\rho$ , $k\Omega$ cm										
		A	BC = A		`DEF = B			GH1 = C			
1B	Control Concrete slab		15.43		15.19			15.46			
2B	Non-inhibitor		7.14		8.21			9.42			
3B		1	50μm,		250μm,			`350µm,			
	Acardium occidentale 1.		13.3		14.3			14.5			

 Table 3.3 : Mechanical properties of Non-Corroded, Corroded and Coated Beam

s/no	Inhibitor (resin/exudates) and	Yield Stress (N/mm²)									
	controlled sample	Time Intervals after 28 days curing									
		A	В	С	D	Е	F	G	Н	I	
		(7days)	(21days)	(35days)	(49days)	(63days)	(77days)	(91days)	(105 days)	(119 days)	
1	Control Concrete slab	410.4	410.1	410.3	410.0	410.3	410.7	410.0	410.5	410.4	
2	Non-inhibitor	4.10.2	410.0	410.0	410.4	410.0	410.3	410.0	410.3	410.2	

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		150µm,				250μm,			`350µm,		
3	Acardium occidentale l.	4.10.0	410.0	410.9	410.8	410.6	410.9	410.7	410.8	410.9	
				Ave	rage valı	ues Yield	Stress (	N/mm <sup>2</sup> )			
		ABC = A				`DEF = B			GH1 = C		
1C	Control Concrete slab	410.27				410.33			410.3		
2C	Non-inhibitor		410.01			410.23			410.17		
		150μm,				250μm,			`350µm,		
3C	Acardium occidentale 1.	410.45			410.77			410.8			
					Ultimate strength (N/mm <sup>2)</sup>						
1	Control Concrete slab	564.7	565.6	562.4	562.6	566.8	562.2	565.2	562.7	562.4	
2	Non-inhibitor	584.7	585.8	586.8	582.8	586.8	582.8	585.4	582.6	588.4	
			150µm,		250µm,			`350µт,			
3	Acardium occidentale 1.	567.7	562.8	562.9	569.8	567.1	563.8	562.1	563.8	564.4	
	Average value (				Average value of Ultimate strength (N/mm <sup>2)</sup>						
			ABC = A			`DEF = B			GH1 = C		
1D	Control Concrete slab	564.23			563.87			563.43			
2D	Non-inhibitor		585.77			584.13			585.47		
			150µm,		250μm,			`350µm,			
3D	Acardium occidentale 1.	564.47			566.9			563.43			

	ISSN 2229-5518			We	sight Lass	nt Loss of Steel Loss (in grams)						
			_	,	San 2000 of oreer 2000 (in grains)							
1	Control Concrete slab	7.25	7.37	7.33	7.25	7.26	7.45	7.28	7.18	7.35		
2	Non-inhibitor	10.628	10.796	10.839	10.876	10.882	10.884	10.835	10.885	10.676		
			150µm,	•		250μm,	•		`350µm,			
3	Acardium occidentale 1.	7.21	7.23	7.29	7.24	7.29	7.32	7.24	7.18	7.27		
			Average valu			eight Los	s of Stee	l Loss (in	grams)			
		ABC = A				`DEF = B		GH1 = C				
1E	Control Concrete slab	7.32			7.33			7.27				
2E	Non-inhibitor	10.754			10.681			10.799				
			150µm,			250μm,			`350µm,			
3E	Acardium occidentale l.		7.24			7.28			7.23			
				Cro	ss- section A	s- section Area Reduction ( Diameter, mm)						
1	Control Concrete slab	12	12	12	12	12	12	12	12	12		
2	Non-inhibitor	11.53	11.53	11.54	11.61	11.64	11.71	11.75	11.76	11.79		
			150µm,		250μm,			`350µm,				
3	Acardium occidentale 1.	12	12	12	12	12	12	12	12	12		
			A	verage Valu	ues of Cross- section Area Reduction ( Diameter, mm)							
			ABC = A		`DEF = B			GH1 = C				
1F	Control Concrete slab	12			12			12				

2F	Non-inhibitor	11.587	11.563	11.662
		150μm,	250μm,	`350μm,
3F	Acardium occidentale 1.	12	12	12

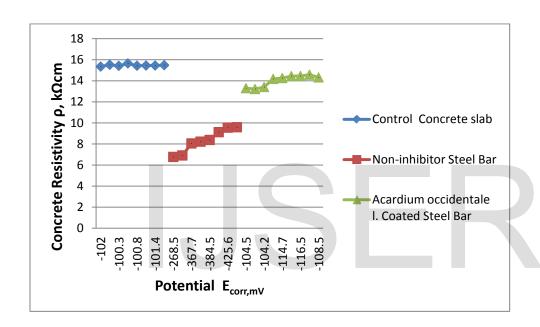


Figure 3.1: Concrete Resistivity versus Potential Relationship Concrete Resistivity  $\rho$ , k $\Omega$ cm versus Potential  $E_{corr,}^{mV}$  Relationship

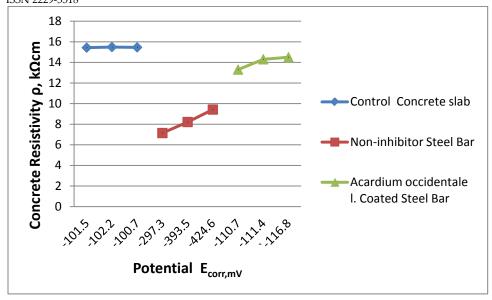


Figure 3.2: Average Concrete Resistivity versus Potential Relationship

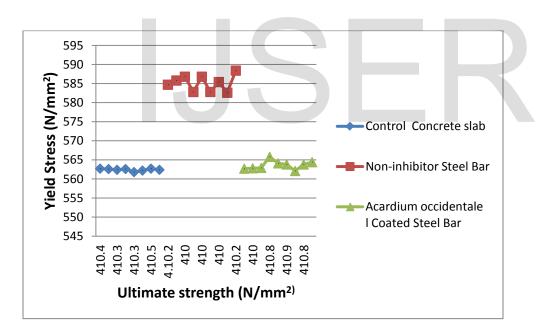


Figure 3.3: Yield Stress versus Ultimate strength.

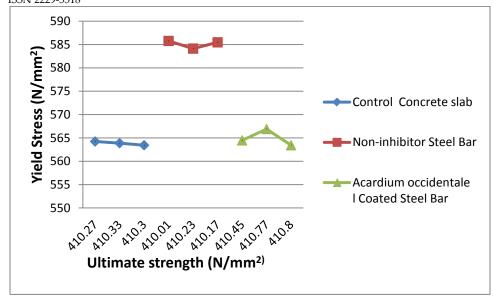


Figure 3.4: Average Yield Stress versus Ultimate strength.

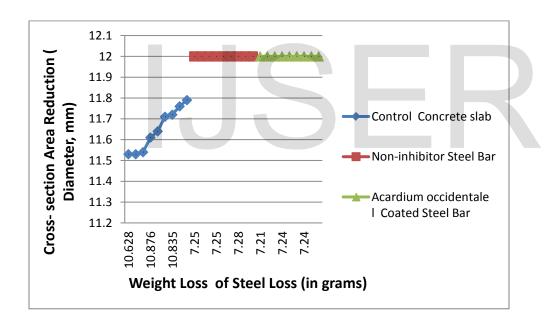


Figure 3.5: Weight Loss of Steel Loss versus Cross-section Area Reduction

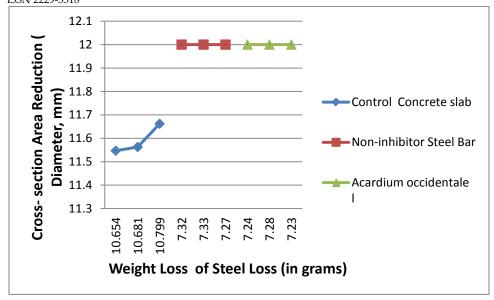


Figure 3.6: Average Weight Loss of Steel Loss versus Cross-section Area Reduction

## 4.0 Conclusion

Experimental results showed the following conclusions:

- i. Entire results showed lower percentages in corroded and higher in coated members.
- ii. Results justified the effect of corrosion on the strength capacity of corroded and coated members.
- iii. Entire results showed higher values tensile strength values in non-corroded and coated to corroded specimens.
- iv. Corrosion potential witnessed in corroded specimens.
- v. Coating thicknesses has influence on corrosion level protection.

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