Electrochemical Potential Investigation of Inhibited Reinforcement Properties Embedded in Concrete in Accelerated Corrosive Medium

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Abstract

The level of chloride attacks on reinforced concrete structures built within the coastal region of Niger Delta of Nigeria has been in alarming rate which has resulted to un-numbered rate of structural failures. The study investigated corrosion level probability assessment potential through half cell potential corrosion measurement, concrete resistivity test and tensile strength test mechanical properties of non-corroded, corroded and inhibited reinforcement with Moringa Oleifera lam resin paste of trees extract. Specimens were embedded in concrete and accelerated in corrosive environment medium for 119 days with required constant current for polarization potential test of -200 mV through 1200mV, with a scan rate of 1mV/s. Results recorded of potential $E_{corr,mV}$, concrete resistivity and tensile strength of moringa oleifera lam 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 $E_{corr,mV}$, and concrete resistivity are 29.9% and 68.74% respectively. When compared to corroded samples, corroded has 70.1% increased values potential $E_{corr,mV}$ and 35.5% decreased values of concrete resistivity. Results of computed percentile average values of yield stress against ultimate strength, when compared to corrode as 100% nominal yield stress decremented from 105.75 % to 96.12% and weight loss at 67.5% against 48.5% and 48.34% to 94.82%, 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

Chlorides have been known to be introduced in concrete through several sources (Morris *et al.* [1] , Ann and Song [2]). Chlorides can be cast into concrete using accelerator agents containing chloride ions, use seawater for concrete mixing and aggregates containing chlorides. Chloride ingress from the environment can be due to seawater entering the concrete structure, ground water with high chloride concentration and deicing salts.

The most important source of chloride ions in concrete is deicing salts which are used in cold climate countries during winter time. The salt mixture penetrates concrete by different mechanisms. These include diffusion which is movement of substance due to a concentration gradient, permeation which is the flow of liquid in concrete due to pressure, capillary absorption which is the transport of liquid into porous non-saturated concrete due to surface tension forces, and migration which is the transport of ions in an electrolyte due to an electrical potential gradient (Mangat and Limbachiya [3], Erdogdu *et al*. [4]).

Diffusion is considered to be the principal form of chloride transport through concrete in aqueous condition. Different factors influence the rate of diffusion of chloride through concrete such as the water/cement ratio, type of cementations material used pore size and distribution, temperature and time (Song *et al.* [5]).

The main factors considered when dealing with diffusion in concrete are pore sizes and distribution in concrete, since the pores filled with water, considered to be the medium which the ions travel through. It has been found (Frey *et al.* [6], Song *et al*. [5]) that concrete samples with high water/cement ratios have a higher diffusion rate than samples with lower water/cement ratios. This has been attributed to the higher volume of macrospores and unsegmented capillary pores present in concrete with high water/cement ratios. Low water/cement ratio can resist chlorides penetration into reinforcing steel, also provides a

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barrier against the entry of oxygen and therefore, provides better concrete corrosion resistance (Canul and Castro [7] , Chia *et al*. [8] , Du and Folliard [9]). The permeability of concrete is a key factor in determining the durability of reinforced concrete structure (Goto and Roy [10] , Guneyisi *et al.* [11]).

Neville [12] estimated that the typical diffusion rates in fully saturated hydrated cement paste to be about 10 – 12 meter square per second, which is so small that it would require several months for the chloride ions to penetrate a 10mm thick hydrated cement paste layer, showing the importance of concrete cover thickness and quality.

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 [13]

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 [14]

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 [15]

2.1.4 Structural Steel Reinforcement

The reinforcements are gotten directly from the market in Port Harcourt. [16]

2.1.5 Corrosion Inhibitors (Resins / Exudates) Moringa Oleifera lam

The study inhibitor Moringa Oleifera lam 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 remore 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 moringa oleifera lam paste were polished and allowed to dried 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 105 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

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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 [17] 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 [18]). Corrosion rates have been related to electrochemical measurements based on data first reported by Stern and Geary [19]. 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 [20] and Langford and Broomfield [21]. 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.

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Table 2.1: Dependence between potential and corrosion probability

Potential E_{corr}	Probability of corrosion
Ecorr < −350mV	Greater than 90% probability that reinforcing steel corrosion is occurring in that area at the time of measurement
−350mV ≤ Ec$_{orr}$ ≤ −200mV	Corrosion activity of the reinforcing steel in that area is uncertain
E_{corr} > −200mV	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 ρ, kΩcm	**Probability of corrosion**
$\rho < 5$	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_{corr} if low (< -350mV) in an area measuring indicates a 95% probability of corrosion. In the other measuring points, potential Ecorr is high (-350mV $\leq E$corr ≤ -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

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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% of 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 3.3 are the results summary and of average values derived from randomly slab samples from A-I of control, corroded and coated specimens of 150µm, 250µm, 350µm summarized to A, B and C from ABC, DEF and GHI. Figures 3.1 and 3.2 are the plots representations of Concrete Resistivity ρ, 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, results showed that non-corroded specimens have 100.3% and 104 .50%, while figures 3.5 and 3.6 are the plots of weight loss versus 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 for non- inhibited concrete specimens on the mapping areas for the accelerated periods of 7days to 119 days which indicated 95% probability of corrosion and indicating a high or moderate probability of corrosion. Average results on comparison showed an increase of 70.1% against 27.2% non-corroded of Potential $E_{corr,}^{mV}$ and 87.8% to 38.8% a decrease values in Concrete Resistivity. Figures 3.1 and 3.2 are the plots representations of Concrete Resistivity ρ, kΩcm versus Potential $E_{corr,}^{mV}$ Relationship. Figures 3.3 and 3.4 are the plots of yield stress against ultimate strength at summary and average state of corroded slab with nominal values of 100% and decreased in ultimate strength from 100.68% to 96.12%, while figures 3.5 and 3.6 presented the weight loss versus cross-section diameter reduction decreased due to attack from sodium chloride from 67.1% to 48.5% and 98.2% to 94.82% respectively.

3.3 Moringa Oleifera lam Steel Bar Coated Concrete Cube Members

Tables 3.1 , 3.2 and 3.3 are the results recorded of potential $E_{corr,mV}$, concrete resistivity and tensile strength of Moringa Oleifera lam inhibited 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 29.9% and 68.74% respectively. When compared to corroded samples, corroded has 70.1% increased values potential $E_{corr,mV}$ and 35.5% decreased values of concrete resistivity . Figures 3.1 and 3.2 are the plots representations of Concrete Resistivity ρ, kΩcm versus Potential $E_{corr,}^{mV}$ Relationship. Figures 3.3 and 3.5 are the plots for arbitrarily and computed percentile average values of yield stress against ultimate strength, when compared to corrode as 100% nominal yield stress decremented from 105.75 % to 96.12% and figures 3.5 and 3.6 respectively presented weight loss at 67.5% against 48.5% and 48.34% to 94.82%, cross-sectional diameter reduction, both showed decreased values of corroded compared to coated specimens.

Table 3.1 : Potential $E_{corr,}$ after 28 days curing and 119 days acceleration Ponding

s/no	Inhibitor (resin/exudates) and controlled sample	**Potential $E_{corr,mV}$**								
		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	Moringa Oleifera lam	-119	-129.5	-124.6	-127.6	-123.6	-127.5	-124.4	-115.5	-111.7
Average values Potential $E_{corr,mV}$										
		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	Moringa Oleifera lam	-124.4	-126.2	`-117.2

Table 3.2 : Results of Concrete Resistivity ρ, kΩcm Time Intervals after 28 days curing curing and 119 days acceleration ponding

s/no	Inhibitor (resin/exudates) and controlled sample	Concrete Resistivity ρ, kΩcm								
		Time Intervals after 28 days curing								
		A (7days)	B (21days)	C (35days)	D (49days)	E (63days)	F (77days)	G (91days)	H (105 days)	I (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	Moringa Oleifera lam	13.18	13.21	13.33	13.59	14.18	14.23	14.32	14.38	13.33

Average values Concrete Resistivity ρ, kΩcm				
		ABC = 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		150µm,	250µm,	`350µm,

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	Moringa Oleifera lam	13.2	13.4	13.64

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

s/no	Inhibitor (resin/exudates) and controlled sample	Yield Stress (N/mm^2)								
		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	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
		150µm,			**250µm,**			**`350µm,**		
3	Moringa Oleifera lam	410.0	410.0	410.9	410.8	410.6	410.9	410.7	410.8	410.9
		Average values Yield Stress (N/mm^2)								
		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	Moringa Oleifera	410.45			410.77			410.8		

	lam									
		Ultimate strength (N/mm$^{2)}$								
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,**			**`350µm,**		
3	Moringa Oleifera lam	567.7	562.8	562.9	569.8	567.1	563.8	562.1	563.8	564.4
	Average value of Ultimate strength (N/mm$^{2)}$									
		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	Moringa Oleifera lam	564/47			566.9			563.43		
		Weight Loss of Steel Loss (in grams)								
1	Control Concrete slab	7.25	7.37	7.25	7.26	7.35	7.28	7.28	7.28	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	Moringa Oleifera lam	7.21	7.23	7.29	7.24	7.29	7.32	7.24	7.18	7.27
		Average values of Weight Loss of Steel 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	Moringa Oleifera lam	7.24			7.28			7.23		
		Cross- 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	Moringa Oleifera lam	12	12	12	12	12	12	12	12	12
		Average Values 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	Moringa Oleifera lam	12			12			12		

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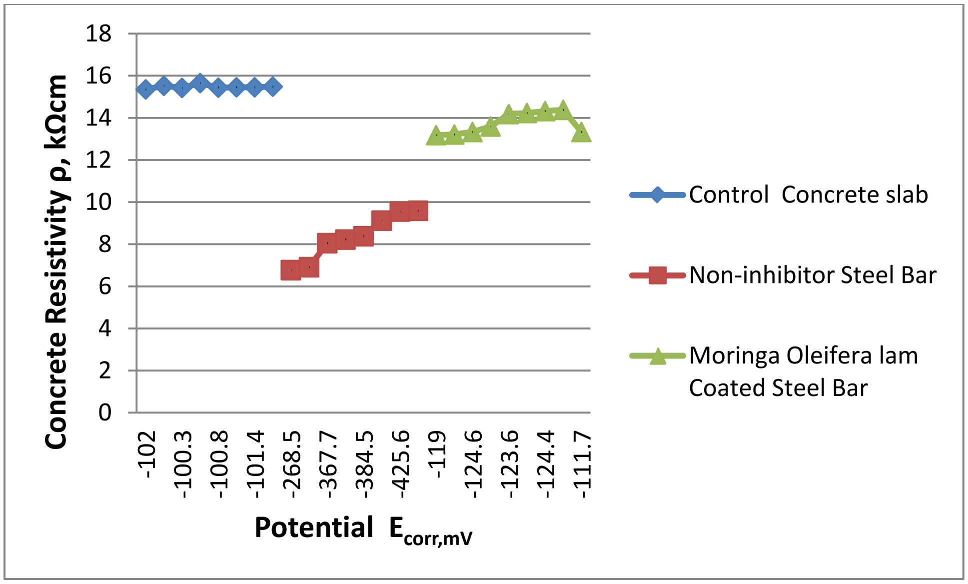

Figure 3.1: Concrete Resistivity versus Potential Relationship Concrete Resistivity ρ, kΩcm versus Potential $E_{corr,}{}^{mV}$ Relationship

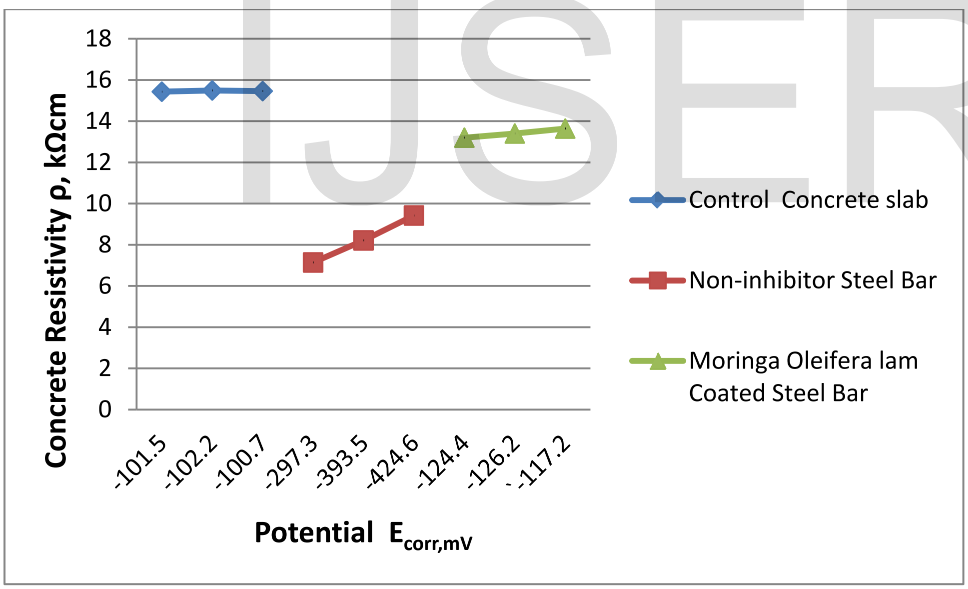

Figure 3.2: Average Concrete Resistivity versus Potential Relationship

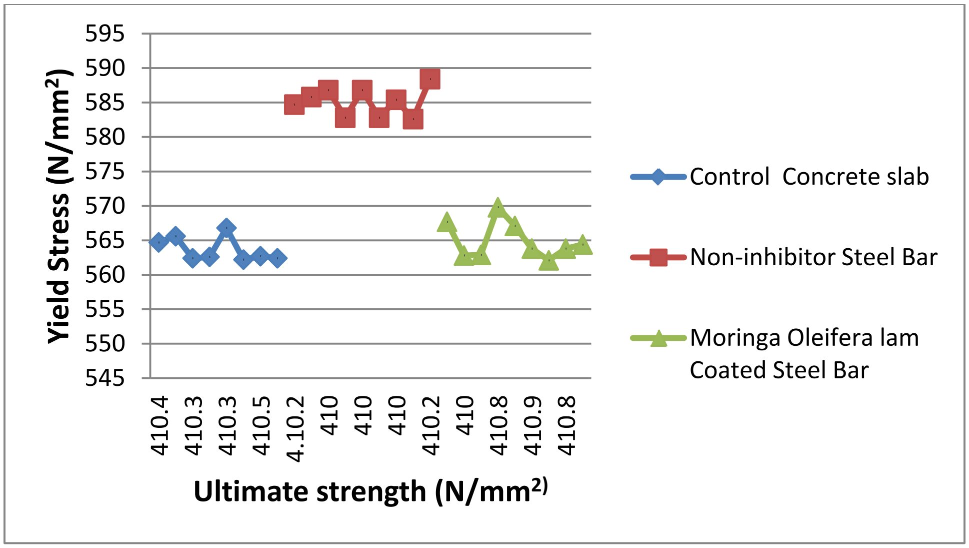

Figure 3.3: Yield Stress versus Ultimate strength

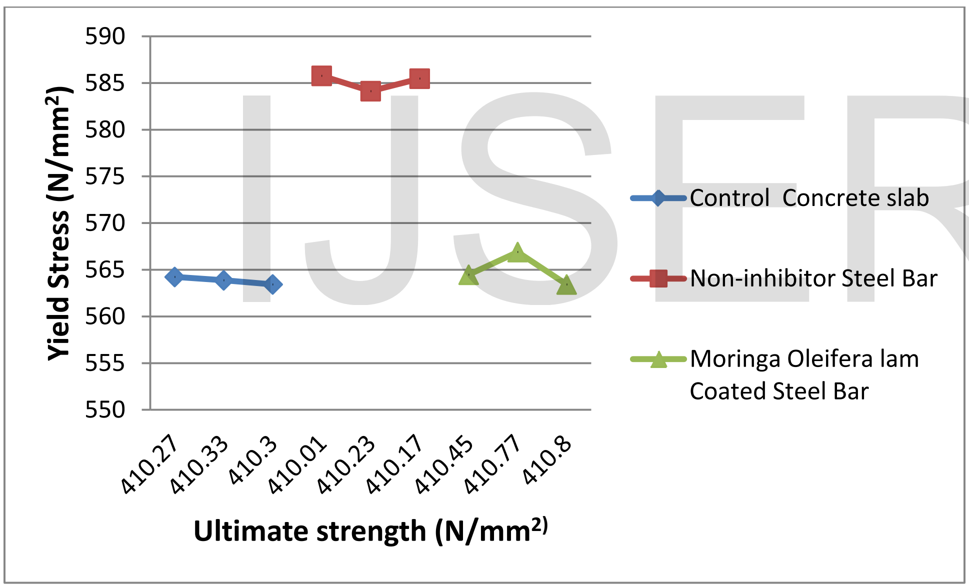

Figure 3.4: Average Yield Stress versus Ultimate strength.

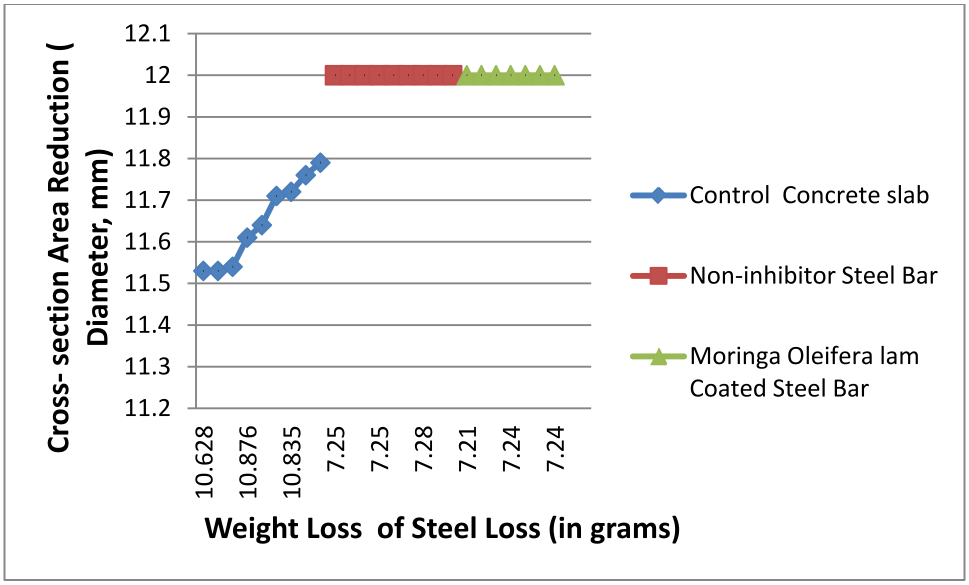

Figure 3.5: Weight Loss of Steel Loss versus Cross- section Area Reduction

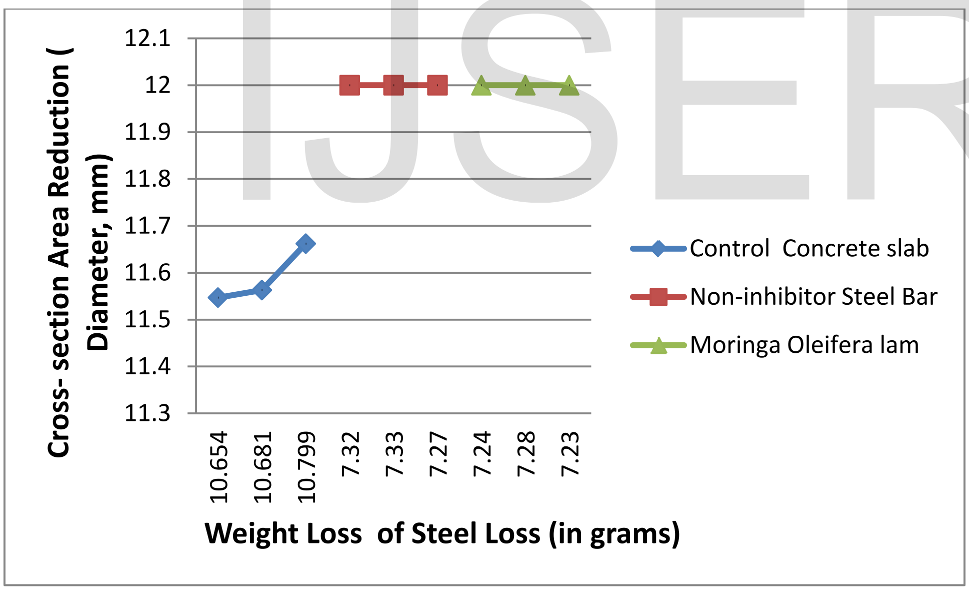

Figure 3.6: Average Weight Loss of Steel Loss versus Cross- section Area Reduction

4.0 Conclusion

Experimental results showed the following conclusions:

i. Corrosion potential manifested on corroded reinforcing steel.

ii. Results justified the effect of corrosion on the strength capacity of corroded and coated members.

iii. Inhibited specimens showed high level of protection against corroded

iv. Inhibitors protect the steel reinforcement against surface demage.

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