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Evaluation of Mechanical Properties of Inhibited Reinforcing Steel in Accelerated Corrosion Medium and Applied Currents Potential

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Abstract

This study investigated the effectiveness in utilization of resins extracts of inorganic inhibitor of ficus glumosa in to curb and prevent the action of corrosion using an experimental study of half cell potential, concrete resistivity and tensile strength and evaluated the change in the surface condition of reinforcements mechanical properties of non-inhibited and inhibited specimens embedded in concrete slab in an accelerated corrosive medium of after 119 days immersion in sodium chloride and with applied currents potential of -200 mV through 1200mV, with a scan rate of 1mV/s. Results recorded of 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. The results recorded of potential $E_{corr,mV}$, and concrete resistivity of ficus glumosa 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 23.75% and 66.48% respectively. When compared to corroded samples, corroded has 73.5% increased values potential $E_{corr,mV}$ and 35.35% decreased values of concrete resistivity, yield stress against ultimate strength at summary and average state of corroded slab with nominal values of 100% and decremented in ultimate strength from 112.48% to 89.25%, weight loss versus cross-section diameter

reduction decreased due to attack from sodium chloride from 69.5% to 47.29% and 48.95% to 77.89% respectively.

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

1.0 Introduction

The rate of corrosion provides information on local corrosive conditions and on the best remedial action to acheive the most effective corrosion prevention. Corrosion measurements can provide early warning of damage in process that result in corrosion induced failure. Determining corrosion rate by measuring weight loss of samples is still in use because it is simple and effective in some situations. However, weight loss only gives an average corrosion of an entire metal sample over the entire test period. The less corrosive medium the longer it would take to get a meaningful test result. Linear polarization resistance (LRP) and potentiodynamic polarization curve measurements are the main electrochemical techniques used to evaluate corrosion rates. Corrosion inhibitors are widely used to delay corrosion of reinforcing steel in concrete. They are chemical substances added to cement or applied to steel reinforcement as epoxy which when properly used, are effective in retarding the corrosion of reinforcing steel in concrete (Gaidis and Rosenberg [1] , Hansson *et al.* [2] , and Justnes [3]). Corrosion inhibitor acts by forming an impervious film on the metal surface or by interfering with either the anodic or cathodic reactions, or both of them. Some inhibitors such as chromates and benzoates have been shown (Ormellese *et al.* [4] , Soylev *et al.* [5]) to reduce the corrosion rate of steel bar, however, but they also reduce the compressive strength of concrete. Corrosion inhibitors can be divided into three general groups: anodic, cathodic, and adsorption inhibitors (Gaidis [6]).

Anodic inhibitors react with the ions of the corroding metals increasing the polarization of the anode and producing thin passive film or salt layers which coat the anode.

Two types of anodic corrosion inhibitors are important for steel: oxidizing agents such as nitrates, nitrites, and chromates, and the other types which require dissolved oxygen so as to be effective such as silicates, phosphates and borates (Justnes [3] , Dhouibi *et al.* [7] , Gaidis [3]).

Cathodic inhibitors affect cathodic reactions. They react with the hydroxyl ions to precipitate insoluble compounds on the cathode site and prevent access of oxygen salts such as zinc, magnesium and calcium or form a layer of adsorbed hydrogen on the cathode surface such as arsenic, bismuth and some organic compounds (Gaidis [3]).

Adsorption inhibitors are adsorbed from the metal surface. These are long organic molecules with side chains which can limit the diffusion of oxygen to the surface, trap the metal ions on the surface, and reduce the rate of dissolution or stabilize the double layer (Gaidis [3]).

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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 [8]

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 [9]

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 [10]

2.1.4 Structural Steel Reinforcement

The reinforcements are gotten directly from the market in Port Harcourt. [11]

2.1.5 Corrosion Inhibitors (Resins / Exudates) Ficus glumosa

The study inhibitor ficus glumosa 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 Mangifera indica 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 119days 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

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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 [12] 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 [13]). Corrosion rates have been related to electrochemical measurements based on data first reported by Stern and Geary [14]. 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 [15] and Langford and Broomfield [16]. 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

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time are assumed.

Table 2.1: Dependence between potential and corrosion probability

Potential E_{corr}	Probability of corrosion
$E\text{corr} < -350\text{mV}$	Greater than 90% probability that reinforcing steel corrosion is occurring in that area at the time of measurement
$-350\text{mV} \leq Ec_{orr} \leq -200\text{mV}$	Corrosion activity of the reinforcing steel in that area is uncertain
$E_{corr} > -200\text{mV}$	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 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 E_{corr} is high ($-350\text{mV} \leq E_{corr} \leq -200\text{mV}$), 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.

Tables 3.1, 3.2 and tables 3.3 are the results 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. Figure 3.3 and 3.4 are the plots of yield stress and ultimate strength of mechanical properties of non-corroded specimens at 100.3% and 100.68%, 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 and tensile strength properties 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 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 Ficus glumosa Steel Bar Coated Concrete Cube Members

Tables 3.1 and 3.2 are the results recorded of potential $E_{corr,mV}$, and concrete resistivity of ficus glumosa 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 23.75% and 66.48% respectively. When compared to corroded samples, corroded has 73.5% increased values potential $E_{corr,mV}$ and 35.35% 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 3.4 are the plots of yield stress against ultimate strength at summary and average state of corroded slab with nominal values of 100% and decremented in ultimate strength from 112.48% to 89.25%, while figures 3.5 and 3.6 presented the weight loss versus cross-section diameter reduction decreased due to attack from sodium chloride from 69.5% to 47.29% and 48.95% to 77.89% respectively

Table 3.1 : Potential $E_{corr,}$ after 28b 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	Ficus glumosa	-124.78	-122.45	-129.98	-125.15	-122.09	-129.46	-124.38	-128.15	-124.75
Average values Potential $E_{corr,mV}$										
		ABC = A			`DEF = B			GH1 = C		

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1A	Control Concrete slab	-101.5	-102.2	-100.7
2A	Non-inhibitor	-297.3	-393.5	-424.6
		150µm,	250µm,	`350µm,
3A	Ficus glumosa	-125.7	-125.9	`-125.95

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	Ficus glumosa	14.02	14.17	14.45	14.58	14.27	14.56	14.51	14.66	14.69

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

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3B		150µm,	250µm,	`350µm,
	Ficus glumosa	14.1	14.5	14.8

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	410.3	410.8	410.9	410.1	410.0	410.5	410.8	410.1	410.3
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	Ficus glumosa	410.6	410.2	410.7	410.7	410.7	410.4	410.6	410.7	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	Ficus glumosa	410.50			410.60			410.77		
		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	Ficus glumosa	565.4	564.7	563.4	565.8	565.8	565.8	568.5	565.45	566.7
	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	Ficus glumosa	564.5			565.8			566.08		
		Weight Loss of Steel Loss (in grams)								
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	Ficus glumosa	7.24	7.25	7.26	7.25	7.29	7.25	7.29	7.25	7.28
		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	Ficus glumosa	7.27			7.26			7.27		

		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	Ficus glumosa	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	Ficus glumosa	12			12			12		

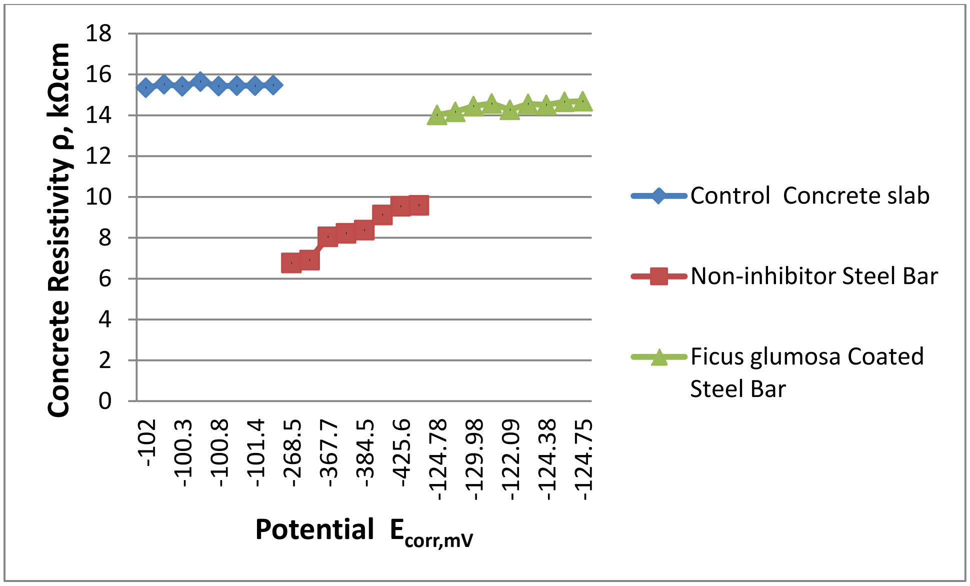

Figure 3.1: Concrete Resistivity versus Potential Relationship Concrete Resistivity ρ, kΩcm versus Potential $E_{corr,}{}^{mV}$ Relationship

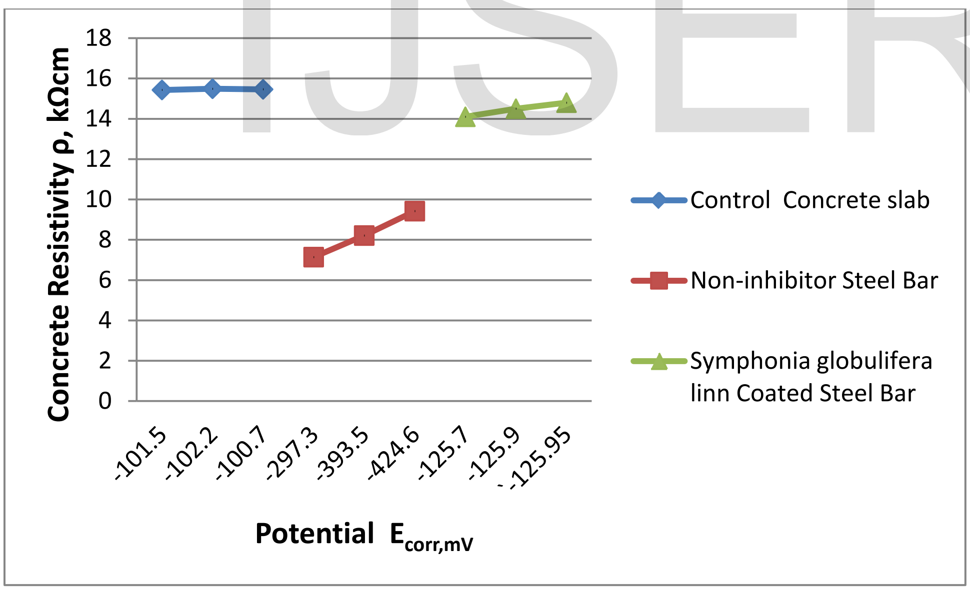

Figure 3.2: Average Concrete Resistivity versus Potential Relationship

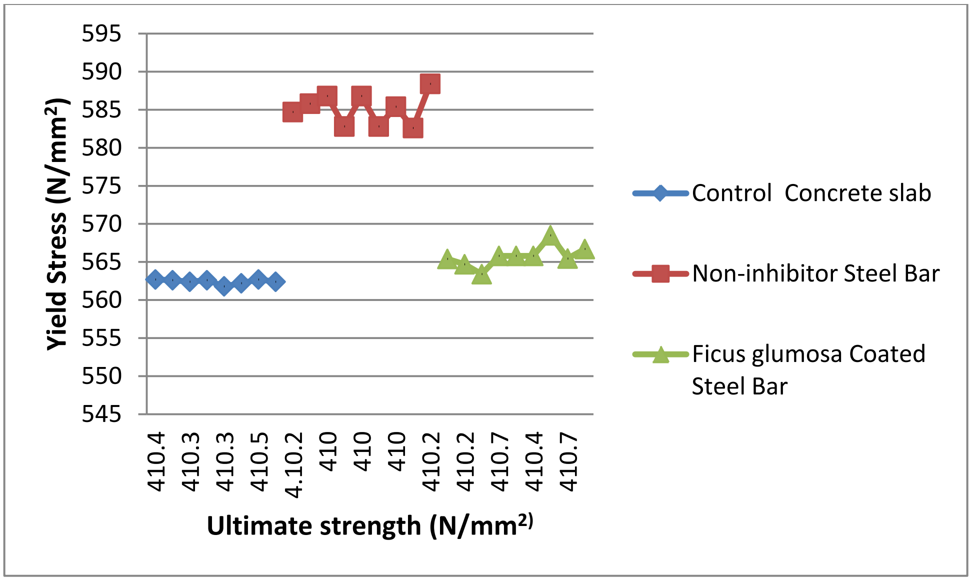

Figure 3.3: Yield Stress versus Ultimate strength

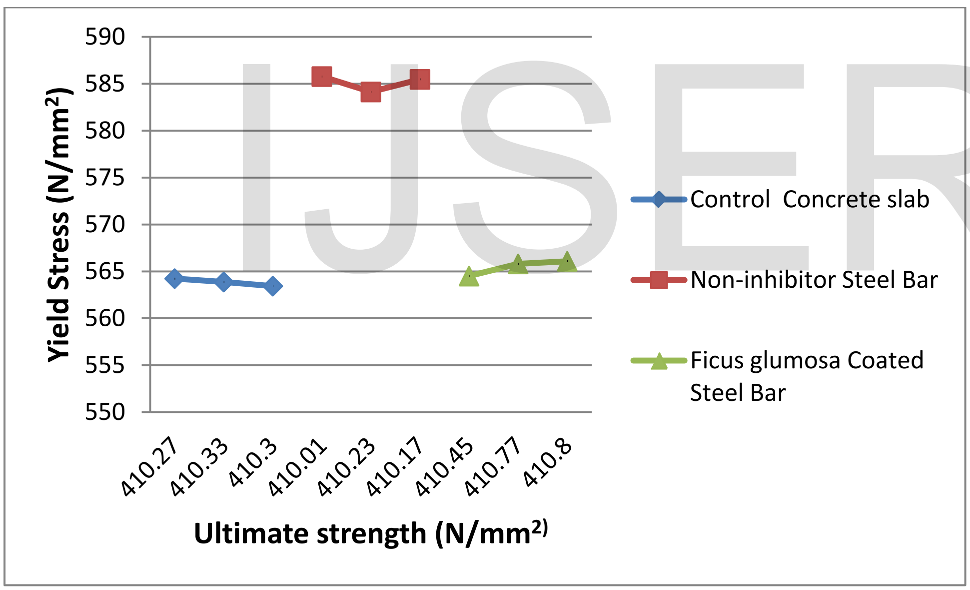

Figure 3.4: Average Yield Stress versus Ultimate strength.

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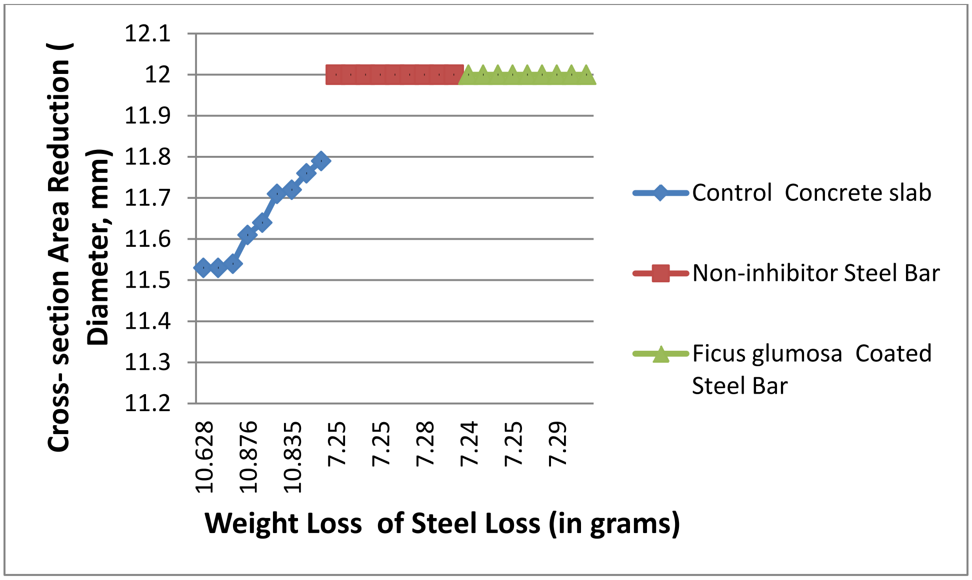

Figure 3.5: Weight Loss of Steel Loss versus Cross- section Area Reduction

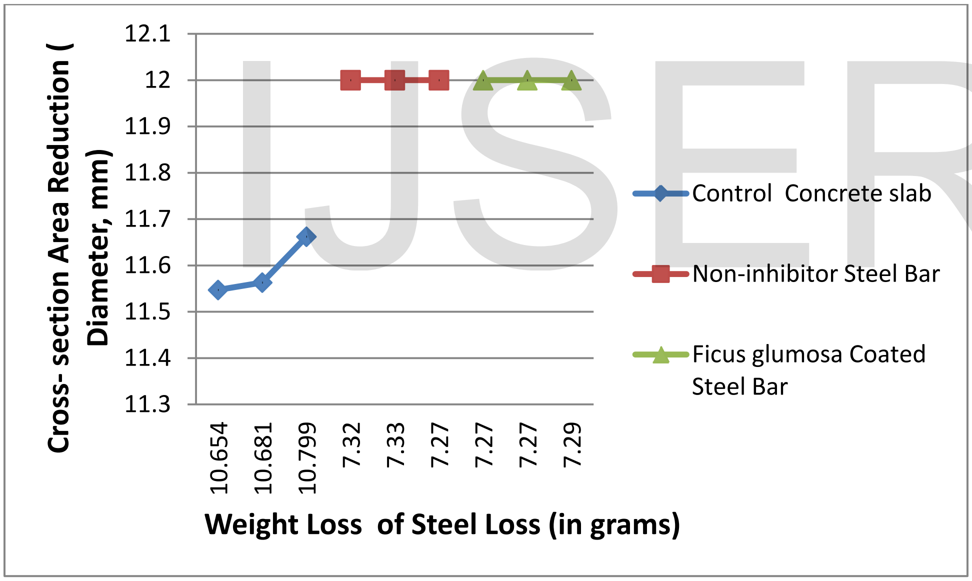

Figure 3.5: Weight Loss of Steel Loss versus Cross- section Area Reduction

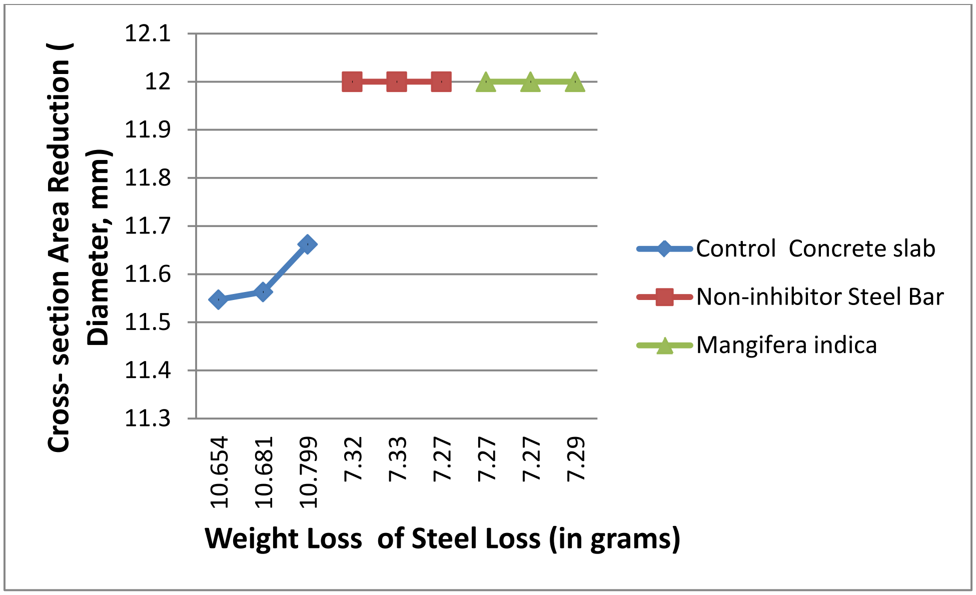

Figure 3.6: Average Weight Loss of Steel Loss versus Cross- section Area Reduction

4.0 Conclusion

Experimental results showed the following conclusions:

i. Potential of corrosion probability was notice on mapping areas

ii. Resin extracts of inorganic origins were discovered to curb and prevent corrosion attack on steel reinforcement

iii. Higher tensile values were obtained from non-corroded and coated compared to corroded specimens

iv. Cracks, spalling and pittings were noticed in non-inhibited specimen.

v. Resins acts as protective coating to reinforcement

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