Study of Sugar Cane Wax Inhibitor on Concrete Reinforcement Corrosion in 3.5% NaCl Solution

A. B. Fajobi, [2]S. O. Akinlolu[1] and O. O. Ige[3]

[1]Department of Civil Engineering, Obafemi Awolowo University, Ile-Ife, NIGERIA
[2]Department of Civil Engineering, The Federal Polytechnic, Ado-Ekiti, NIGERIA
[3]Department of Materials Science and Engineering, Obafemi Awolowo University, Ile-Ife, NIGERIA
afajobi@yahoo.com; afajobi@oauife.edu.ng; ige4usa@yahoo.com

Abstract: This study explored the effect of sugar cane wax on rebar corrosion in chloride contaminated solution with a view to determining its impact on the compressive strength of the inhibited reinforced concrete. The medium carbon steel (8 mm diameter and 100 mm length) was embedded in a concrete with mix ratio 1:2:4 and water:cement of 0.65. 42 concrete cubes were cured for 21 and 28 days, whereby some samples were subjected to 3.5% NaCl solution with and without inhibitor; the inhibitor concentration varied between 1 – 5%. Compressive strength test was used to examine the mechanical behavior and the corrosion measurements were evaluated using weight loss and potentiostatic polarization techniques. The compressive strength test revealed a reduction in the concrete strength as the inhibitor concentration increases. The optimum inhibitor efficiency was recorded at 5% for both potentiostatic polarization and weight loss method. The experimental data were best fitted with Langmuir adsorption isotherm which indicated chemisorption of the inhibitor monolayer on the steel surface. In conclusion, sugarcane wax was found to protect the reinforcing bars effectively despite the high deterioration of the compressive strength of the rebar.

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1. Introduction

Millions of dollars are lost each year because of corrosion in construction industry. Much of this loss is due to the corrosion of iron and steel, although many other metals may corrode as well. Damages from this corrosion initiates when salts, water and air penetrate through the pores of concrete and reach the surface of the steel. In the past, most of the design studies in the literature and research in reinforced concrete assumed that the durability of reinforced concrete structures could be taken for granted. However, many reinforced concrete structures are exposed during their lifetimes to environmental stress (for example, corrosion and expansive aggregate reactions) which attacks the concrete or steel reinforcement (Ormellese *et al.*, 2009). Researchers and engineers are continuously in search of cost-effective means to prevent the corrosion of reinforcing steel for the duration of a concrete structure's design life. The cement paste in concrete is alkaline with a pH typically between 12 and 14. This paste forms a passive film surrounding reinforcing steel in concrete which further thicken iron oxide layer on the steel surface. Many researchers believe this alkaline environment facilitates the protective passive film around the steel (Broomfield, 2003; Soeda and Ichimura, 2003; Gaidis, 2004; Ormellese *et al.*, 2006; Ormellese *et al.*, 2009). The passive film is not invulnerable, though it can be damaged both chemically and mechanically. Some examples of chemical damage are carbonation, chloride ingress (seawater, de-icing salt, unwashed sea sand, admixtures etc) and sulphate attack. Proper design and preparation of concrete in accordance with relevant standards and timely maintenance of the structures under those conditions would guarantee them a long and efficient life in aggressive media. However, these requirements are not always met and adhered to. Preventive measures being used in the construction industry to salvage the service life of steel reinforcement in concrete structures are cathodic protection, inhibitors, coatings, penetrating sealers and chloride removal (Saraswathy and Song, 2007). One of the practiced methods popularly used for the control of steel corrosion in concrete is the corrosion inhibitors either preventive or curative.

An ideal inhibitor would be a compound preventing corrosion without unfavourable effects on the properties of concrete and also without environmental hazards. Generally, the inhibitors used in concrete are inorganic compounds such as phosphates, chromates, nitrites and nitrates, although some of them have toxic effects and pollute the environment. Organic inhibitors often considered as possible substitutes, are based on amines or organic acids. It is well known that the presence of an organic molecule in the corrosive media inhibits corrosion of metals by adsorbing at the metal-solution interface. The investigation of nature-designed molecules and blends of naturally synthesized chemical compounds as possible inhibitor candidates for reinforcing steel in concrete is of raising interest due to the health and environmental hazards involved in the use of most abiogenic inhibitors. This work considers the application of sugarcane wax as corrosion inhibitors for rebar corrosion.

Sugarcane wax is a whitish to dark yellowish extracted from sugarcane juice and it is only one of the several coproducts of sugarcane and is about 0.1% with chemical signature of complex and variable mixtures. The mixtures consist of long chain alkenes, hydrocarbons, fatty acids, ketones, aldehydes, alcohols and esters, and steroids such as β-sitosterol, stigmasterol, ketosteroids and hydroxyl keto steroids [Bhosale *et al.*, 2012; Inarkar and Lele, 2012]. This work is conceptualized due to the beauty of sugarcane wax which can be attributed to its economic sustainability by encouraging the waste to wealth philosophy popularly advocated by developing countries. Also, the health, safety and environmental sustainability are predicated on the biogenic origin of the products. A handful of researchers have stated that sugarcane wax have widespread applications in cosmetics, paper coating, textiles, fruit and vegetable coating, leather sizing, lubricants, adhesives, polishes, and pharmaceutical industry [Georges *et al.*, 2006; Taylor, 2000]. The application of sugarcane products so far in corrosion prevention and control is restricted to sugarcane juice as additives in electroplating process. There are few reports in respect of sugarcane juice extracts and its synergistic effects on zinc plated mild steel [Loto 2012a; 2012b; 2013]. It is noting that patent for nitrogen-containing corrosion inhibitors for metals based on sugarcane wax in oil industry is the only studied that have reported the application of sugarcane extracts as inhibitor (Ledovvskykh *et al.*, 1988).

In view of this the primary objective of this work is to evaluate the application of sugarcane wax as inhibitor for built environment in acidic environment. Furthermore, another purpose of this paper is to highlight the impacts of compressive strength on the inhibited rebar concrete. A review of literature indicated that the implication of curing rate on the corrosion phenomena is not well documented. Hence, this work will elicit information on the corrosion rate as a function of the curing period. The corrosion rates were determined by mass loss and potentiodynamic measurements in 3.5% NaCl environment at room temperature. The research objectives does not include quantification of localized corrosion in the studied environment. A compressive strength test was also performed on the rebar which revealed the effect of the admixed inhibitor on the strength of the concrete.

2. Methodology

2.1 Materials

Medium carbon steel was used for this study since it is the commonly used material for reinforcement in construction. It was obtained commercially and the chemical composition analysis was carried out spectrometrically at Grand Foundry, Lagos, Nigeria with a percent nominal composition of 0.370C, 0.293Si, 0.640Mn, 0.048P, 0.048S, 0.0082Mo, 0.26Cu, and the balance being Fe.

Crushed limestone with maximum size of 6.25 mm from ESPRO Company Nigeria Limited, Wasimi, Ile-Ife was used as coarse aggregate. The sand was obtained from a site located at Obafemi Awolowo University, Ile – Ife, Nigeria having specific gravity (2.64) and average absorption ratio (0.57%) was used as fine aggregate. Elephant Portland cement was used in all the concrete mix and the research environment was 3.5% NaCl. The sugarcane wax is sugar mill solid waste produced by mechanical pressing and the yield varied from 3.5 to 4.1%. It was sourced from open market in Abuja, Nigeria and its concentration in the admixture ranges from 0 – 6% at an interval of 1%.

2.2 Preparation of concrete cubes

42 reinforced concrete cubes with cubes dimension (100 x 100 x 100 mm) and steel bars with 8 mm diameter were used for this research. A clearance of 20 mm was given between the steel and the bottom of the concrete cube. The mix ratio used for this experiment was ***1:2:4*** for cement, sand, and gravel respectively. The concrete batching was done by weight according to the work of Idowu (2012), and each batch consists of six specimens. The mix ingredients in each batch are; Portland cement (1.86 kg), sand (3.72 kg), gravel (7.44 kg), sodium chloride (0.13 kg) and water (1.21 kg) for uninhibited plus sugar cane wax for inhibited environments and the water to cement ratio used was 0.65. The concrete constituents were mixed for both uninhibited and inhibited samples according to the work of Adamu *et al.*, (2014a,b) by mixing in a pan for approximately 3 – 5 minutes until a uniform mixture was achieved. Thereafter, the moulds were filled with concrete in approximately three equal layers and vibrated for consolidation using physical effort. The batching, mixing and casting were performed on a single day for each of the seven batches and the curing (21 and 28 days) was done by covering the finished surface with polyethylene sheet. This implied that all specimens are of the same age and tested at the same time.

2.3 Weight loss measurements

In order to evaluate the weight loss, the steel samples were weighed before being used to reinforce the concrete. The rebar corrosion-cubes were immersed in the environment for a total period of 32 days at an interval of 4 days each. The corroded sample was removed, the concrete-cube opened, the rebar sample cleaned thoroughly, then weighed with the aid of a Mettler weighing balance and recorded to the nearest 0.1 mg. The corrosion rate was then calculated using the formula below (Fontana and Greene, 1996):

$$CR = \frac{534W}{DAT} \quad (1)$$

where, CR is corrosion rate (mpy); W is weight loss of samples (mg); A is area of samples (in^2); T is exposure time (hr); and D is density of medium carbon steel (g/cm^3).

The inhibition efficiency of the inhibitor was calculated using the following expression:

$$IE\% = \frac{100\,(CR_i - CR_f)}{CR_i} \quad (2)$$

where, CR_i and CR_f are the corrosion rate of the steel in the without and with inhibitor respectively.

2.4 Tafel analysis

This adopted the electrochemical measurement procedure as described by Olorunniwo *et al.*, (2015) whereby a three electrode potentiostat was employed. The cell consists of Ag/AgCl as reference electrode, a carbon graphite rod as counter electrode and the reinforced steel in the concrete as the working electrode, while the electrolyte was made up of 3.5% NaCl solution. The open circuit potential (E_{oc}) was measured using a digital multimeter connected to both the rebar and reference electrode. This is achieved within a window of 10 – 30 minutes depending on the period required for the E_{oc} to stabilize. The potentiostat is based on a 10 byte read/ 16byte write I/O system and the polarization scanned the samples from 0 – 1000 mV than the free corrosion potential (Ecorr), at a scan rate of 10 mV/s based on earlier studies (Vasilescu *et al.*, 2010; Sheela *et al.*, 2005 and Mueller and Hornberger, 2014). The Tafel plot was manually extrapolated at the linear proportion of the cathodic curve about 200 mV away from E_{oc} with a vertical line intersecting the corrosion portion of the curve. This enabled the determination of the corrosion current I_{corr} with which the corrosion rate was calculated using ASTM G 102-89 (1999) standard as given below:

$$CR(mpy) = \frac{K_i \, x \, I_{corr} \, x \, EW}{dA} \quad (3)$$

where, CR = corrosion rate(mpy); K_i = 3272 mm/(amp-cm-year); I_{corr} = corrosion current density (μAcm^2); EW = equivalent weight (gm); d = rebar steel density (g/cm^3) ; EW = equivalent weight; and A = exposed sample area (cm^2).

The inhibitor Efficiency was calculated according to equation (2).

2.5 Compressive strength measurement

This test was done by crushing the concrete cube using ELE 1560 KN compressing testing machine with the aim of determining the impact of the sugar cane wax on the strength of the concrete cube. The concrete cube was placed between the plate of the compressive machine and an incremental force was applied until the cube crumbled. The applied load at this stage was noted and this was repeated for the concrete cubes with and without inhibitor for each curing period of 21 and 28 days. The compressive strength (N/mm^2) was then determined using the formula below:

$$\text{Compressive strength} = \frac{Force}{Surface\ area} \quad (4)$$

3. Results and discussion

3.1 Weight loss

The loss of asset integrity noticed for uninhibited samples and the subsequent reduction in the degradation rates as obtained for inhibited environment from weight loss data is presented in this section. The consensus in rebar corrosion research has been that environmental factors most especially the concentration of aggressive species play domineering role in the steel dissolution. Thus, the concept of rebar corrosion can be better appreciated by understanding the theoretical background. Generally, there is a wide accepted postulation that chloride induced reinforced corrosion is ascribed to the formation of hydrogen chloride and chloride ions thereby leading to pH decrease in the concrete environment (West and Hime, 1975). This local acidification of the electrolyte is governed by the following reactions:

$$2Fe + 2Cl_2 = 2FeCl_2 \text{ (unstable)} \quad (5)$$

$$FeCl_2 + 2H_2O = Fe(OH)_2 + 2HCl \quad (6)$$

This process coupled with the presence of medium carbon steel leads to iron dissolution at anode (equation 6), HCl dissociation (equation 7), and consumption of dissolved oxygen and the electrons produced at the anode (equation 8).

$$Fe = Fe^{2+} + 2e^- \quad (7)$$

$$HCl = Cl^- + H^+ \quad (8)$$

$$O_2 + H_2O + 4e^- = 4OH^- \quad (9)$$

The exhaustion of the chlorides and stoppage of HCl production resulted in the release of ferrous hydroxide, $Fe(OH)_2$ or in the presence of surplus oxygen, ferric hydroxide, $Fe(OH)_3$ as the corrosion products.

$$Fe^{2+} + 2(OH)^- = Fe(OH)_2 \quad (10)$$
$$Fe^{3+} + 3(OH)^- = Fe(OH)_3 \quad (11)$$

The presence of this corrosion products are supported by Pourbaix diagram for iron-water system in alkaline medium (Adamu *et al.*, 2014a, b).

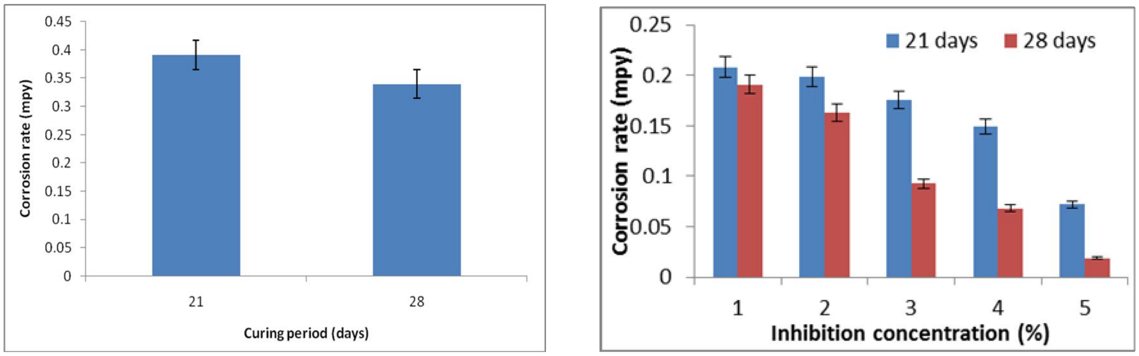
Figure 1a displayed the degradation rates of the uninhibited samples at different curing period and it was observed that a longer curing period (28 days) exhibited better higher corrosion behaviour with sample cured at 21 days. There is 13% improvement in the rebar corrosion rates that is the corrosion rates of 0.34 and 0.39 mpy were observed for 21 and 28 days respectively. It is assumed that since the curing rates have direct consequences on the concrete pores and rheological properties. Similarly, with the addition of sugarcane wax, the results obtained follow the same trend with uninhibited. For example, the materials wastage for inhibited samples at 21 and 28 days were 0.208 and 0.191 mpy respectively with 1% sugarcane wax. A possible explanation is that the inhibitor neutralizes the environmental agents causing the degradation by inducing increase concrete alkalinity thereby preventing breakdown of the protective passive oxide film on the steel surface. The impact of the curing period on the degradation rates becomes significant as the inhibitor concentration increases and obviously, 5% inhibited samples cured for 28 days offers the best corrosion resistance (0.019). While the same set of samples cured for 21 days has corrosion rate of about 74% higher (Figure 1b). It is *suspected* that the sugarcane wax reinforced the durability of the rebar concrete by neutralising the chloride ions thereby ensuring high alkalinity. This allows the formation of a thin stable passive oxide film strongly adherent to the steel surface. It is safe to assume that the film adherence is a function of the sugarcane wax concentration, in that the corrosion behaviour improves as the inhibitor increases until it reaches optimum at 5%. Another plausible explanation for the admixing inhibitor *performance* may be adduced from the chemical reactions point of view. According to Inarkar and Lele (2012), the major component of sugarcane wax is sec-butyl isothiocyanates which is an ester. It has molecular weight of 99.13 and linear molecular formula (C_5H_9NS) which shows that it has nitrogen and sulphur elements. These elements which have been adjudged has having relative good inhibitory properties coupled with the presence of double, triple bonds or aromatic rings which *tend* to form stronger coordination bonds. This is in consonance with the work of Ergün *et al.* (2008) which states that compounds having π-bonds generally exhibit excellent inhibitive properties since the π-orbital will provide the electrons for the surface interaction.

3.2 Tafel plots

A complete quantification of the significance of sugarcane wax as corrosion inhibitor in concrete chloride-induced environment using potentiostatic polarization technique is as presented in Table 1 and Figure 2. Table 1 is the summarized corrosion rate of the samples using potentiostatic technique and noticeably, similar pattern was observed as recorded for gravimetric method. In that, samples cured for longer period have better corrosion protection and using uninhibited samples to demonstrate revealed that the corrosion rates were 0.134 and 0.0601mpy for 21 and 28 days cured samples respectively. Also, the impact of the curing rate becomes pronounced as the inhibitor concentration increased and this is as shown for 5% inhibited samples with 0.047 and 0.0215 mpy for 21 and 28 days cured samples respectively. Literature have established that combining both Tafel and Faraday laws indicates existence of relationship among current density, polarization resistance and corrosion rate. While, both current density and corrosion rate increases, polarization resistance behaves contrarily (Fontana, 1987). In this study, the observations were that both corrosion rate and current density decreased as the inhibitor increases. This demonstrates that the presence of the inhibitor reduces the degradation rate associated with the working electrode. Whereas the E_{oc} values as obtained from Figure 2 were pretty on the high side and this can be attributed to the acidic nature of the environment. Though Tafel analysis were not without some drawbacks which includes the assumption that the extrapolated region were within activation over potential and not accounting for the possibility of having high currents thereby making the corrosion systems under mass transport control.

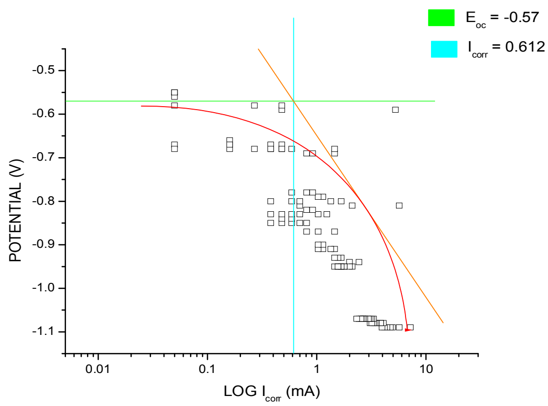
Table 1: Summarized potentiostatic corrosion rate at different curing rate

Inhibitor concentration (%)	Corrosion rate (mpy)	
	21 days	28 days
0	0.134	0.0601
1	0.128	0.0533
2	0.1235	0.0469
3	0.113	0.0444
4	0.096	0.0261
5	0.047	0.0215

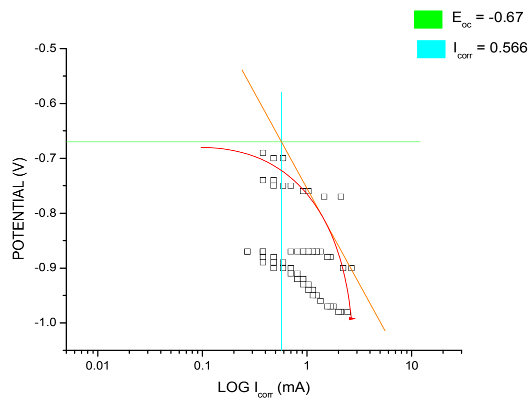


a b

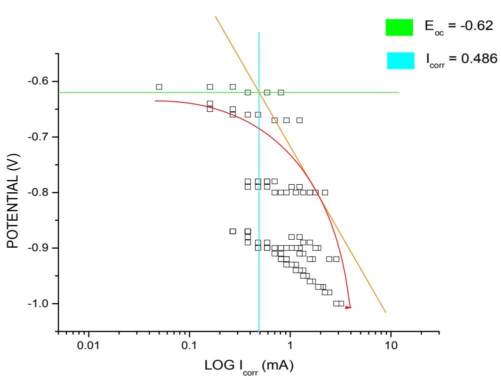
Fig. 1: The corrosion rate for (a) uninhibited and (b) inhibited samples at different curing times (21 and 28 days) by using weight loss method



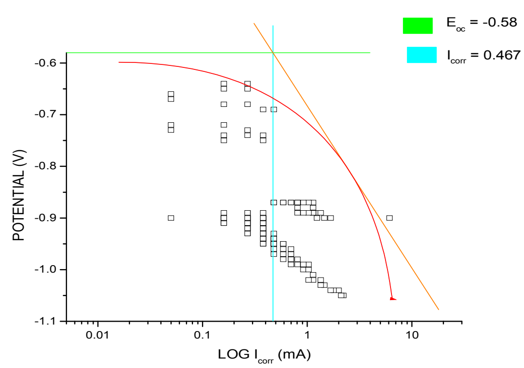
(a)



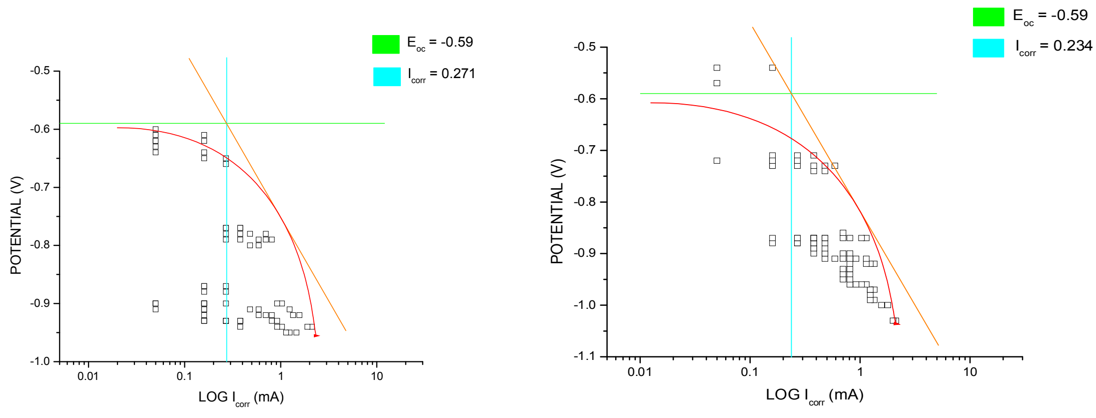
(b)



(c) 2%



(d) 3%



(e) (f)

Figure 2: A plot of voltage versus Log of current (a) uninhibited (b) 1% (c) 2% (d) 3% (e) 4% and (f) 5% inhibited sample

Notwithstanding, this work favour this methodology mainly because it is easier to measure experimentally and the assumption of carrying out the extrapolation the linear portion of the cathodic polarization 200 mV below E_{oc} to the measured corrosion potential (Ningshen and Mudali, 2002; Sequira, 2010). The second assumption was render nullify by the fact that although the region was outside activation control region but in majority of the plots, the activation control region does not display the required linearity over an order of magnitude of current (Olorunniwo *et al.* 2015).

3.3 Inhibition Efficiencies

Fig. 3 shows a plot of inhibitor efficiency versus inhibitor concentration after curing for different period (21 and 28 days) using potentiostatic polarization method. The efficiency increased as the inhibitor concentration increases which are in line with previous studies until the optimum value is obtained [Adamu *et al.*, 2014a, b]. This increment can be attributed to the effectiveness in the sugarcane wax to modify the chloride induced ions and the resultant steel/environment surface properties. Infact, Adamu *et al.*, (2014) argued that the improved performance is the ability of inhibitor complex ions to arrest the chloride ions. The optimum for this study occurred at 5% after which the efficiency decreases from preliminary investigation. In concrete technology, reduction in pore spaces are associated with prolonged curing period thereby reducing chloride ions penetration into the reinforced concrete. Consequently, higher inhibitor efficiency was recorded after curing for 28 days compared to 21 days since lower amount of aggressive ions will be exposed to the reinforced steel bar.

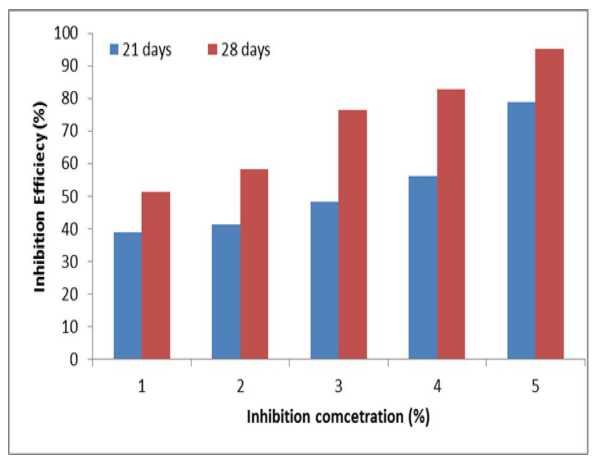


Figure 3: Inhibition efficiency of the sugar cane wax at different curing rates using potentiostatic method

3.4 Compressive strength

The inhibitor concentrations have polynomial relationship with the compressive strength and even the corrosion rate (Figure 4). The principle of this relationship is that of mass action kinetics or chemical reaction systems which implies that the corrosion rate reaction is proportional to the corrosion products concentrations of the participating molecules. In order words the reaction rates are proportional to the reactive species which also assumed that the corrosion reactions are considered as a series of elementary reactions. Finally, sugarcane wax being an organic inhibitor type is expected to be governed by surface chemistry reactions controlled by the limiting current. The average compressive strength for the concrete without sugar cane wax was 12.2 N/mm^2 after curing at 21 and

28 days. This implies that the curing period does not have any distinct effect on the compressive strength for uninhibited samples. Fig. 5 shows a plot of average compressive strength versus inhibitor concentration after curing for 21 and 28 days. This shows a slight increase in the compressive strength for those specimens that underwent 28 days curing period. This is also as a result of reduced pore spaces with increase in curing period. This graph also shows a drastic reduction in the compressive strength of the specimens with addition of the inhibitor. A cursory examination of the addition of sugarcane wax has negative impacts on the reinforced concrete cubes compared with uninhibited, for example a dramatic reduction to about 22% was noticed with 1% sugarcane wax. There is no clear cut standard in the literature while some reported improvement in the addition of inhibitors to the compressive strength, other studied reported drastic adverse effect of the inhibitors in rebar corrosion vis-à-vis compressive strength. Also, noticeable is that there is no correlation of this behavior with inhibitor types; be it, anodic/cathodic, organic/inorganic among others (Loto, 1992). The only *inference* is that the introduction of sugarcane wax in reinforced concrete will results in *loss* of compressive strength probably due to weak inhibitor chemical reaction hardening *setting* with the concrete. This negativity trend continues as the inhibitor concentration increases until it reaches around 4% whereby it stabilizes around 1.2 N/mm^2. This value was recorded with addition of 5% sugarcane wax which corresponds to the optimum inhibitor concentration noticeable. It can be stated that the inhibitor concentration and its impact have reached threshold at this point. This adverse effect implies that additives must be added to compensate for the comprehensive strength of the sugarcane wax.

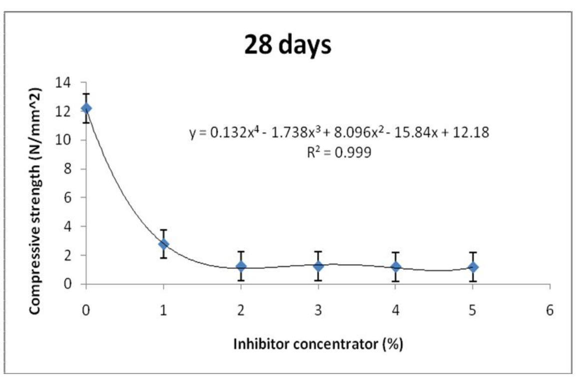


Fig. 4: Effects of inhibitor concentration on the compressive strength of the reinforced concrete at different curing periods (21 and 28 days)

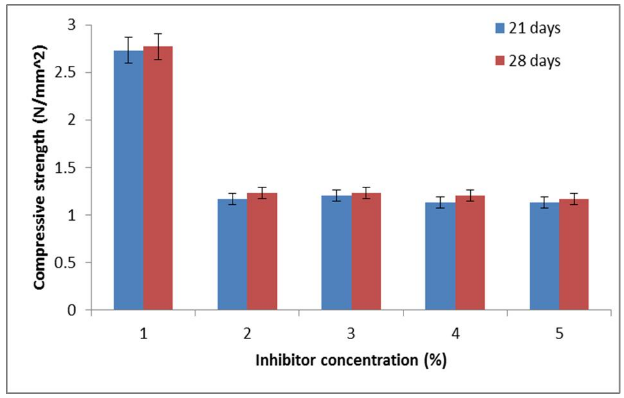


Figure 5: A typical graphical representation showing the relationship between compressive strength and inhibitor concentrations

4. Conclusion

- Sugarcane wax is suitable as corrosion inhibitors in built environment with 5% being the optimum concentration
- Addition of sugarcane wax has pronounced deteriorative impact on the reinforced concrete compressive strength hence; it is recommended that a complimentary additive must be determined to compensate for the reduction in its compressive strength.
- Increasing the curing period have beneficial effects on the corrosion behaviour of the reinforced concrete in the presence of sugarcane wax and there is negligible impact on its compressive strength.

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