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Corrosion Behaviour of Heat Treated and Nickel Plated Mild Steel in Citrus Fruit: Lime and Lemon

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Abstract: Corrosion behavior of heat treated and nickel plated mild steel exposed to citrus solutions (Lime and Lemon) was investigated in this study. Annealing, normalizing, hardening and tempering heat treatment were performed on mild steel, and their effects on microstructure of the mild steel were studied. Corrosion analysis of untreated, heat treated, nickel plated on heat treated samples of mild steel in the citrus solutions was done using potentiodynamic polarization technique. Results revealed that the use of nickel as electroplating element and heat treatment were quite effective when used as a means of resisting corrosion of mild steel. Heat treated mild steel samples had better corrosion resistance in citrus solution than untreated sample with a corrosion rate of 1.88080 mmpy. Furthermore, Nickel plated tempered mild steel substrate has a superior corrosion resistance in lemon which correlated with the performance of samples when exposed to lime environment with corrosion rate of 0.10853 mmpy. Although, some of the nickel plated samples on heat treated mild steel substrates exhibit high corrosion rate in lemon, but plated hardened substrate sample was found to have the highest rate of 1.76350 mmpy. Plated annealed substrate sample also exhibits a good corrosion resistance with a corrosion rate of 0.81645 mmpy. In addition, plated normalized substrate sample exhibits the highest corrosion rate in lime solution with corrosion rate of 1.7492 mmpy, whereas plated annealed substrate sample has a corrosion resistance with the rate of 0.60474 mmpy.

Keywords: Mild Steel, Heat Treatment, Nickel Plated, Citrus Fruit, Corrosion, Microstructure.

I. INTRODUCTION

Low carbon steel is the least expensive type of steel and commonly used for construction purposes due to its availability, low cost and good mechanical properties, but it is highly susceptible to corrosion, especially when exposed to atmospheric oxygen in a wet environment[1].Due to the high corrosion of carbon steel, different investigations have be conducted to reveal the corrosion characteristics of steel in different environments such as HCl, H_2SO_4, NaOH, processed water, rain water, municipal tap water, cassava extract, cocoa extract etc. [2-6]. Furthermore, heat treatment of low carbon steel has been used to enhance ductility, toughness, strength and hardness which are very good for structural applications and additionally, to relieve internal stress developed in the material[7-12]. Also, application and effects of heat treatment on corrosion of mild steel have gained attention of some authors. Babata et al.[5]studied corrosion behaviour of commercial mild steel in municipal tap water. The mild steel was subjected to different heat treatments (annealing, hardening, normalizing and tempering) and the results revealed the heat treated samples had better corrosion resistivity than untreated (as received) sample, most especially the annealed and normalized samples.

Igwemezie and Ovri[13] studied the effects of micro structural change and corrosion susceptibility of heat treated medium carbon steel in different corrosive media. The study showed that microstructures obtained by different heat treatment processes are sensitive to the environment and corrosion as a result of ferrite precipitation and cementite phases. Uncoated low carbon steel is unsuitable for use in food processing, particularly in wet environment, because of relatively high corrosion rate. Studies have been conducted on the possibility of plating these steels with nickel through electrode position and assess their corrosion behaviour, and suitability in food media. Oloruntoba et al.[14]investigated the effect of plating parameters (current density, bath concentration and solution volume, and electroplating time) on nickel electroplating of low carbon steel. The results of their findings generally showed that increase in any of these variables will increase the thickness of nickel electro deposition on low carbon steel.Corrosion behaviour of nickel plated low carbon steel in tomato fluid was investigated by Oluwole and Olawale [4].Thick nickel coating was observed to offer protection in tomato fluid environment. Oluwole et al.[15]studied corrosion resistance of nickel-plated medium carbon steel and 18/8 stainless steel in cyanide environment (cassava fluid).

Un-plated steel was found to have very high corrosion rate whereas 18/8 stainless steel was found suitable for use in this environment. The renewed corrosion activity on nickel plated steel after the 20th day (pH = 12) of continuous use in cyanide environment makes it unsuitable for use. Momoh et al.[16]also studied the corrosion performance of nickel electroplating of heat treated low carbon steel substrate in Na_2CO_3 environment. It was reported that the rate of corrosion of nickel plated tempered low carbon steel substrates is significantly low compared to the non-plated sample. Food processing equipment demands highly clean and sanitized look, strength and corrosion resistance, making stainless steels the material of choice in this regard. However, in recent times in Nigeria, there has been an increased in the local fabrication of food processing equipment using alternative indigenous materials which are not stainless steels in order to minimize cost without compromising the quality of these foods processing equipment. These materials are low and medium carbon steels (coated and uncoated), galvanized steels etc. Low carbon steel is readily available and cheaper compared to stainless steel. It can easily be modified through heat treatment for structural applications, and by hard coating can serves the same purpose of stainless steel without undermining quality expected of foods processing equipment. This research therefore focuses on the surface modification of heat-treated low carbon steel by the process of electrode position of nickel and evaluate it corrosion resistance in citric environment using potentiodynamic polarization electrochemical method.

II. MATERIALS AND METHODOLOGY

A. Materials

The mild steel used in this research work was purchased from a local market in Ado-Ekiti, Nigeria. Nickel powder used for the plating was acquired from Winteck, Ikeja, Lagos. Citrus fruits (lime and lemon) were purchased from King's market in Ado-Ekiti and Isinkan market in Akure, Nigeria. The juice of the fruits were obtained by manual squeezing and filtered with a sieve, and then stored in a covered and labeled jars at room temperature prior to use. The mild steel of 5 mm thickness was mechanically cut into square shape coupon of dimension 10 mm × 10 mm. The elemental composition of the mild steel (Table 1) was determined using Arc Spark Spectrometer prior to heat treatment procedures.

Table I: Chemical Composition of the Low Carbon Steel

Elements	C	Al	S	P	Mn	Ni	Cr	As
Composition	0.1700	0.0018	0.0500	0.0400	1.0300	0.200	0.500	0.0007
Elements	Mo	V	Cu	Si	Zn	Sn	Ca	Fe
Composition	0.060	0.045	0.2	0.280	0.0045	0.020	0.003	97.4

B. Heat Treatment

The heat treatment procedures were performed in an electric muffle furnace (Figure 1), capable of attaining temperature exceeding 1200^{o}C. Sixteen samples of the cut mild steel were selected for the heat treatment, four samples for each of the heat treatment process, while four samples were left as-received.

1. Annealing

Four of the selected samples were first heated to a temperature of 900^{o}C to obtain austenite phase and later held for 1 hour at that upper critical temperature to enable the samples at that temperature have sufficient time for proper homogenization. The furnace was then switched off so that the samples temperature will decrease with the same rate as that of the furnace (furnace cooling). The samples were taken out of the furnace after a day when the furnace temperature had already reached the room temperature.

2. Normalizing

The four selected samples for normalizing were first heated to austenitic phase at a temperature of 900^{o}C and kept at this temperature for an hour for proper homogenization. The furnace was then switched off and the samples were taken out and air cool to room temperature.

3. Hardening

The samples selected for hardening were heated to the temperature of 900℃in the muffle furnace, allowed to homogenize for an hour, before the heated samples were quenched in water bath to ambient temperature, was taken out of the bath and cleaned properly.

4. Tempering

The specimens for the tempering were heated to 900℃ for an hour and then quenched in the water bath maintained at room temperature; the quenched samples were then reheated to a temperature of 230℃.

Fig 1: Muffle furnace.

C. Microstructural Examination (Optical Micrograph)

Cut samples for the microstructural test were progressively grinded to obtain a smooth and flat surface using different grits emery paper in decreasing coarseness, and polished metallographically using polishing cloth and paste with alumina particles until a mirror-like surface was obtained. Thereafter, the mirror-like polished surface samples were etched in 2% Nitric acid and 98% Ethyl alcohol to reveal the microstructure of the surface layer. The phases of the specimens were then photographically recorded at 100 and 200 magnifications using Zeiss metallurgical microscope.

D. Electroplating Process

1. Degreasing

The specimens to be electroplated (two specimens from each of the heat treatment processes and two as-received) were first degreased in a degreasing chamber (Figure 2(a))to remove adhering grease and oil stain on the surface of the material. Degreasing solution was prepared by hydrolysis method. 10g of sodium hydroxide was mixed with 30g of sodium carbonate (ratio 1:3). The add-mix was charged into a 5 litres bath containing 2 liters of deionized water. The bath was fitted with a thermometer and heated to 60^{o}C to obtain a clear solution. The heat treated samples (one specimen from each of the heat treatment process and one as-received) were introduced into the prepared solution, and degreased by gently swirled while maintaining the temperature at 60^{o}C for 2 hours. The process was stopped and the specimens withdrawn into a cold swirl to remove alkaline traces from the surface of the materials. The specimens were washed with excess deionized water and dried over silica gel for 12 hours for the moisture to be removed.

2. Nickel Plating

Prior to the nickel plating of the degreased specimens, the specimens were cleaned dried with a cotton wool and weighed using analytical electronic weighing balance (FA2204B) to obtain their initial weights. Plating was carried out in a previously prepared nickel electrolyte; 250g of Nickel chloride, 100g of Nickel sulfate, 5g of Boric acid and 25 litres of deionized water.

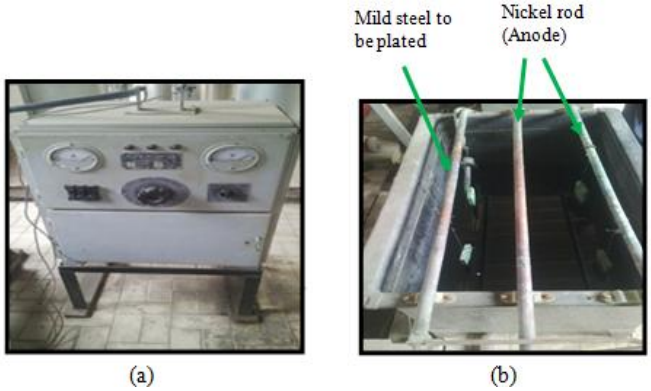


Fig2: (a) Greasing chamber and (b) Setup of the plating bath process.

The plating bath was fitted with heater and a temperature probe,the cathode on which the samples were hung was sandwiched by two sacrificial anodes each carrying two nickel bars of dimensions 20cm × 5 cm totally immersed in the electrolyte. Direct current of 2 volts was passed through electrolyte thereby resulting into the transfer (oxidation) of ions from the sacrificial anode (nickel) to the cathode (mild steel) (reduction) as deposits (coating). The temperature was maintained at 50^{o}C and the plating time was 35 minutes. After plating, each specimen was drawn into a cold swirl, rinse with excess deionized water and dried over silica gel for 12 hours. The samples were then finally weighed after plating process. Figure 2(b) shows the experimental setup of the plating process.

E. Corrosion Tests

The corrosion behaviour of the nickel plated-heat treated substrate samples was investigated in lime orange and lemon orange solutions (pH 2.40 and 2.30 respectively) at room temperature (25^{0}C) using potentiodynamic polarization electrochemical method in accordance with ASTM G59-97 (2014). The experiments were performed using a three-electrode corrosion cell set-up comprising the specimen as the working electrode, saturated silver/silver chloride as reference electrode, and platinum as counter electrode. Working electrodes shown in Figure 3(a) were prepared by attaching an insulated copper wire to one face of the sample using aluminum conducting tape, and cold mounting it in resin after which the sample surfaces were polished progressively with emery papers starting from 120grit to 640grit size. The samples were degreased with acetone and then rinsed in distilled water before immersion in the prepared solutions of lime and lemon orange solution respectively, exposed to atmospheric air. The working electrodes were immersed in test solutions, as depicted in the experimental set up shown in Figure 3(b), until a stable open circuit potential was obtained. Open circuit potential measurements were carried out in separate cell for 120 minutes. Potentiodynamic polarization measurements were carried out using a scan rate of 0.16 mV/ s at a potential initiated at -200 mV to +250 mV. After each experiment, the electrolyte and the test samples were replaced. The results of the corrosion tests were evaluated by Tafel plot extrapolations to determine the corrosion current densities (Icorr), corrosion potentials (Ecorr) and corrosion rate.

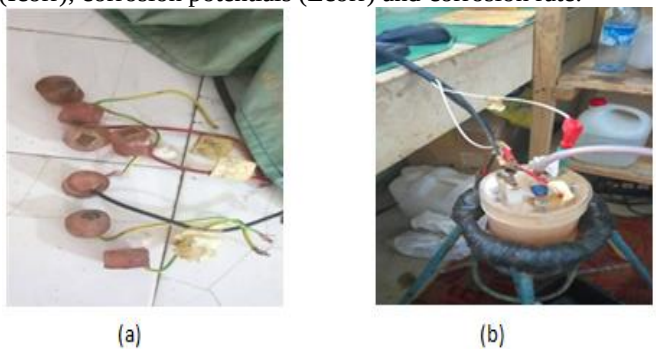


Fig 3: (a) Working electrodes and (b) Experimental set up for electrochemical process.

III. RESULTS AND DISCUSSION

A. Micro structural Evaluation

Figure 4 shows the optical micrographs of the as-received and heat treated samples. The microstructure of the as-received mild steel (Figure 4(a))composed of ferrite identified by the white patches in the grain boundaries of the acicular pearlite grains (the dark patches), and this is also reported by Igwemezie and Ovri [13]. Consequently, the microstructure of the as-received mild steel can be described as having a ferrite-austenite phase. However, tempering heat treatment (Figure 4(b)) resulted into formation of tempered marten site microstructure which is a double phase mixture of low-carbon marten site and Ɛ-carbide as also reported by Clover et al.,[17].Annealing heat treatment of mild steel affected the spatial distribution of ferrite at the grain

boundaries of the micro structure as shown Figure 4(c). It can also be seen that scales were present in the ferrite, which might be due to oxidation at the metal surface, and this was also stated by Joseph et al.[18].

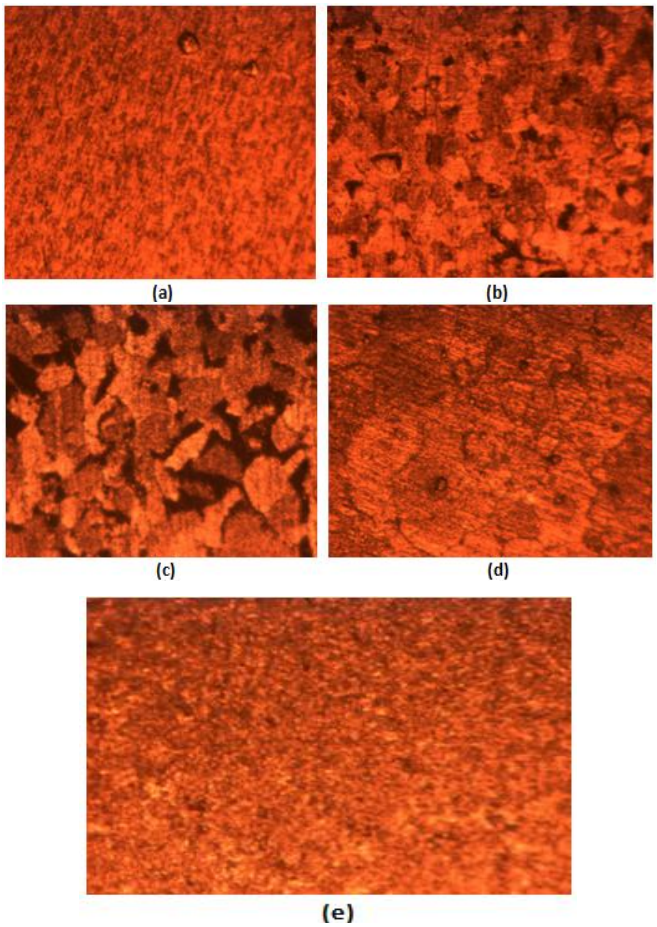


Fig 4: Micrograph showing (a) as-received (b) tempered (c) annealed (d) normalized (e) hardened mild steel microstructures (× 200 magnifications).

In this microstructure, the slow cooling condition in the furnace (annealing) allowed large amount of carbon diffusion resulting into considerable precipitation of ferrite. Hence, giving rise to an equiaxed grains of ferrite and pearlite as reported by Igwemezie and Ovri [13]. In contrast, normalized mild steel sample (Figure 4.1 (c))showed a more uniform fine grained microstructure of ferrite and pearlite with large grain sizes. This was due to rapid method of cooling in air unlike slow cooling in furnace (annealing), and this retarded ferrite grain growth witnessed in the matrix of pearlite. Furthermore, hardened mild steel as shown Figure 4(e) indicates the presence of dispersed ferrite within a cementite distribution (carbide) in martensite matrix, and this in in conformity with the reported by Igwemezie and Ovri [13].

B. Plating Evaluation

The results of the measured weight before and after electroplating procedures with respect to plating time are shown in Table 2. It can be observed that increase in weight of 0.30g, 0.40g, 0.31g, 0.35g, 0.38g were obtained after electroplating for samples 1 (Control), 2 (Tempered), 3(Annealed), 4 (Normalized) and 5(Hardened), respectively, with respect to plating time. The results of this measured weight show that the tempered and hardened samples have relatively higher deposition, while the control sample has the least weight. The affinity for the plated nickel might responsibly for the significantly low rate of corrosion of nickel plated tempered low carbon steel substrates is significantly low compared to the non-plated sample as reported by Momoh et al.[16].

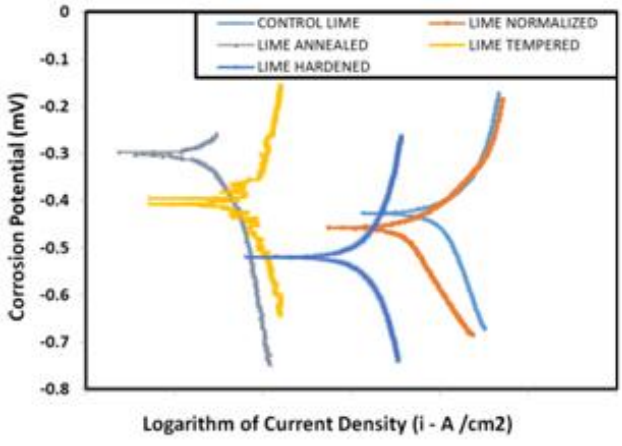
Table II: Samples Weight Before And After Plating with time of Plating

S/N	specimens	Weight before plating (g)	Weight after plating (g)	Weight difference
1	Control	6.30	6.60	0.30
2	Tempered	6.10	6.50	0.40
3	Annealed	6.70	7.01	0.31
4	Normalized	6.20	6.55	0.35
5	Hardened	6.10	6.62	0.38

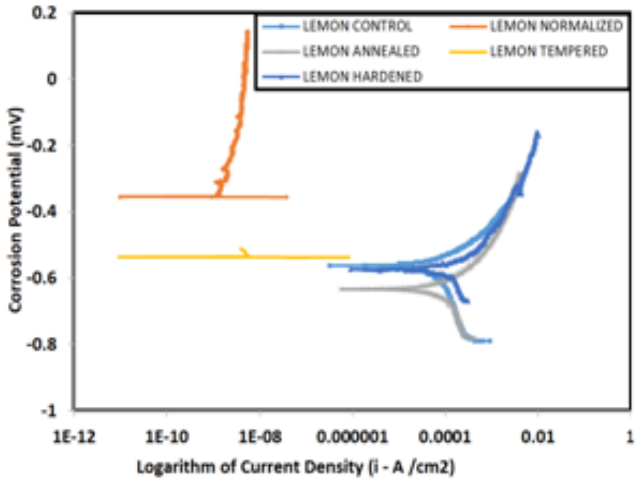
C. Corrosion Evaluation

1. Corrosion behaviour of heat treated samples

Figure 5(a) shows the corrosion results of the as-received and heat treated samples obtained from the Tafel plots corrosion studies in lime environment, while the Tafel extrapolations of the corrosion current densities (I_{corr}) and corrosion potentials (E_{corr}) are presented in Table 3. It can be observed from the results that all the heat treated samples displayed better corrosion resistance when compared with the as-received sample. Nevertheless, the tempered and annealed samples exhibited superior corrosion resistance of 0.0004463 mmpy and 0.0000213 mmpy, respectively. The observed behaviourin the tempered sample could be attributed to the low stress levels in the steel sample which makes the localized breakdown of the passivity more difficult than other structure as stated by Atandaet al.,[19]. The corrosion behaviourexhibited byannealed sample could be due to observed thicker corrosion products covering the surface of the sample, thereby forming a protective film on the surface which inhibited the corrosion rate.



(a)



(b)

Fig 5: Polarization curves of as-received and heat treated samples in (a) lime and (b) lemon.

The corrosion results of tafel plots electrochemical studies and the tafel extrapolations of the corrosion current densities (I_{corr}) and corrosion potentials (E_{corr})in lemon indicate clear distinct corrosion behaviour between the as-received and heat treated samples. It is observed that annealed and hardened samples have corrosion rates of 0.01240 mmpy and 0.52830 mmpy, respectively which are higher than observed value in lime medium. The higher corrosion rates observed in these samples might be due to breakdown of protective film during the period of investigation. However, tempered sample shows similar good performance of low corrosion rate as observed in lime medium with a corrosion rate of 0.00011 mmpy. This behaviour has been attributed to the low stress levels in the steel sample which makes the localized breakdown of the passivity more difficult than other structure [19].However, all the heat treated could be adjudged to perform well in lime and lemon media, except for hardened sample in lemon, their corrosion rates are well below 0.2 mmpy.

Table III: Electrochemical Data of Samples from the Tafel Extrapolations In Lime And Lemon Solution

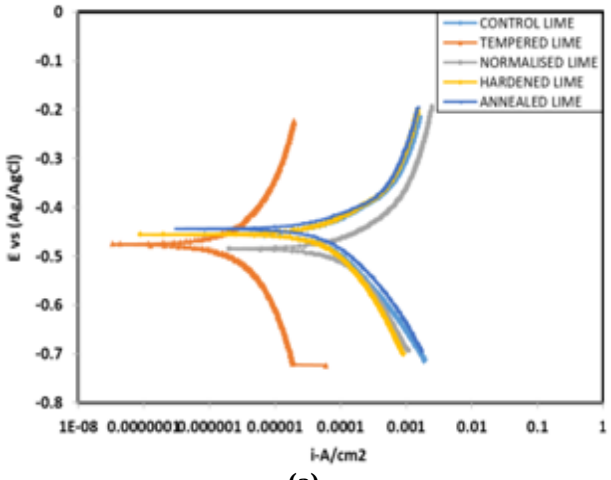
Heat-treated samples	Potential E_{corr}(mV)	Corrosion Current I_{corr} (μA)	Corrosion (mmpy)
		Lime	
Control	-430.2290	162.0830	1.88080
Tempered	-407.6290	38.4630	0.00044
Annealed	-300.9130	1.8350	0.00002
Normalized	-461.8100	16.9620	0.19680
Hardened	-520.2330	11.4900	0.13340
		Lemon	
Control	-563.5000	79.5840	1.92347
Tempered	-450.4560	9.2650	0.00011
Annealed	-633.4200	259.6060	0.01240
Normalized	-461.8100	75.8950	0.14560
Hardened	-573.1520	304.0650	0.52830

2. Corrosion behavior of plated samples

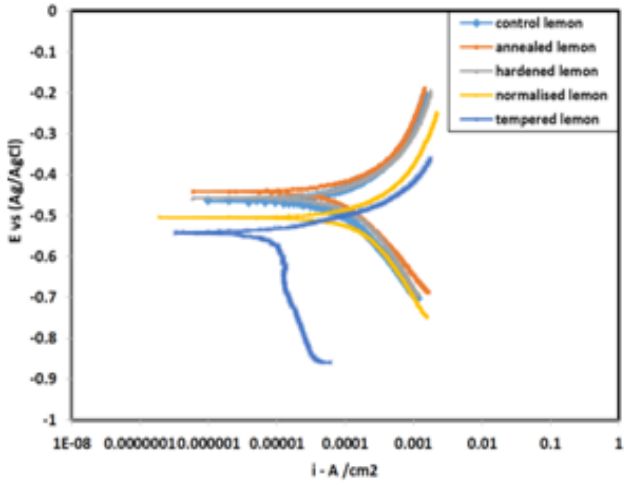
The corrosion results obtained from the tafel plots electrochemical studies in lime and lemon environments (Figure 6 and Table 4) indicate clear distinct corrosion behavior between nickel plated with non-heated and heat treated mild steel substrates. It can be observed from Table 4that plated tempered mild steel having the corrosion rate of 0.05311 mmpy displayed the best corrosion resistance while plated normalized sample exhibited the highest corrosion rate of 1.7492 mmpy. However, plated annealed sample showed a good corrosion resistance with corrosion rate of 0.60474 mmpy while plated as-received (non-heated) and plated hardened sample displayed corrosion rate of 0.8250 mmpy and 0.95281 mmpy respectively. This gives an indication that only plated tempered mild steel is best suited for lime environment. However, the observable differences in corrosion rates could be as a result of ferrite and cementite phases which might led to setting up of galvanic cells within the microstructure with the cementite phase becoming cathodic and the ferrite anodic thereby accelerating corrosion reaction as reported by Igwemezie and Ovri [13] while deposition of nickel might have aided better performance of plated tempered sample as this might increase ferrite electrochemical resistance and preventing the formation of carbide as stated by Cloveret al.[17].

Table IV: Electrochemical Data of Plated Samples From The Tafel Extrapolations In Lime And Lemon Solution

Heat-treated samples	Potential E_{corr}(mV)	Corrosion Current I_{corr} (μA)	Corrosion (mmpy)
		Lime	
Control	-455.6630	-70.7170	1.82058
Tempered	-476.2490	-4.5770	0.05311
Annealed	-443.8170	-52.1160	0.60474
Normalized	-484.9390	-150.7430	1.74920
Hardened	-445.8450	-82.1130	0.95281
		Lemon	
Control	-122.2990	-465.5280	1.49191
Tempered	-10.7090	-541.0960	0.10853
Annealed	-70.3610	-441.5170	0.81645
Normalized	-149.7890	-505.2670	1.73810
Hardened	-151.9700	-456.7480	1.76350



(a)



(b)

Fig6: Polarization curves of nickel plated as-received and heat treated mild steel substrates in (a) lime and (b) lemon.

It can also be observed from Figure 6 that plated tempered sample having the corrosion rate of 0.10853 mmpy exhibited superior corrosion resistance in lemon medium which is in agreement with its performance in the lime environment, while plated hardened sample exhibited the highest corrosion rate of 1.76350 mmpy; this could be as a result of combining effects of ferrite precipitation, residual stress and carbide precipitation which tends to cause higher corrosion rate in the hardened structure even when plated nickel as reported by Igwemezie and Ovri [13]. Plated hardened sample and plated-normalized sample displayed corrosion rate of 1.76350 mmpy and 1.73810 mmpy respectively, which are higher that plated as-received with corrosion rate of 1.49791 mmpy. The observed higher corrosion rates might be due to constant breakdown of protective film during the period of investigation. However, plated annealed sample showed a good corrosion resistance with corrosion rate of 0.81645 mmpy. The presence of nickel might have also contributed to the good corrosion resistance of all the heat treated samples as their corrosion rate were below 2.0 mmpy.

IV. CONCLUSION

Experimental investigation has been carried out on the corrosion behavior of heat treated mild steel in lime and lemon environment, the following conclusions were drawn within the limit of work:

- After heat treatment, as-received showed a ferrite grain in boundary within acicular pearlite while tempered exhibited low carbon tempered marten site. Annealed heat treatment process affect spatial distribution of ferrite in a pearlite matrix. Hardened sample showed a ferrite grain in marten site matrix whereas the normalized sample results into fine distribution of ferrite-pearlite matrix.
- The corrosion results showed that microstructures obtained by different heat treatment processes are sensitive to both lime and lemon corrosive environment, and corrosion of the samples is due to ferrite precipitation and carbide phases.
- Heat treatment of mild steel has also been observed to be beneficial in the two corrosive environment investigated.
- The use of nickel as electroplating element on heat treated and non-heated was observed to be effective on the corrosion behaviour of mild steel.
- Plated and un-plated tempered and annealed samples gave good corrosion behavior with plated tempered having better performance both in lime and lemon environments.
- Heat treatment process and nickel electroplating can be used to control corrosion of mild steel in citrus fruit (lime and lemon) environment.

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