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Effects of Surface Preparation on the Corrosion Behavior of Mild Steel

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A B S T R A C T

Surface preparation of engineering materials is necessary for preventing corrosion and subsequent failure of materials in service. There are different methods of surface preparations that can affect engineering materials in different ways. This study investigated the effect of surface preparation on the corrosion behavior of zinc sprayed and unsprayed mild steel. Quantitative analysis and potentiodynamic polarization techniques were used to evaluate the immersed samples of different surface preparations. The results indicated that the least corrosion rate was observed for the uncoated sample prepared with CC1200 grit paper at 0.041 mpy and successive samples in the order of CC220 grit paper at 0.047 mpy < P60 grit paper at 0.052 mpy < filing at 0.064 mpy and grinding at 0.074 mpy after 42-days of immersion. The prepared samples that were further coated with zinc spray demonstrated a similar trend. The sample prepared with CC1200 grit paper and further coated with zinc spray exhibited the lowest corrosion rate of 1.35×10^{-9} mpy. Potentiodynamic polarization results further suggested that the same behavior was observed in the quantitative analysis.

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1. INTRODUCTION

Corrosion of metals is one of the oldest problems that have ever challenged the industrial world and is defined variously [1]. Corrosion is the gradual physiochemical destruction of materials by the action of the environment. It is also known as the deterioration of materials or their properties because of reaction with its environment. In spite of the findings that are available on corrosion control, prevention, and mitigation, studies are still ongoing on these issues because of the importance of corrosion mitigation. The reasons are not far-fetched due to the fact that the mechanism of corrosion formation and significant effect of factors such as temperature, exposure time, the concentration of the aggressive components, corrosion control constituents, and surface finishing is very dynamic and, therefore, not predictable [2].

Corrosion of mild steel is of great practical interest because it is widely used in the construction of oil, gas, and offshore environments for pipelines, flow lines, platforms, down-hole tubular equipment, wellheads, industrial vessels.... etc. Corrosion inhibition is being extensively employed in minimizing metallic wastage of engineering materials in service [3] and surface coating is one of the methods used in inhibiting or reducing corrosion.

Different engineering processes can be used to enhance the surfaces of some types of steel prior to painting or coating. However, the surface finishing of mild steel, stainless steel, and duplex steel affects the corrosion resistance, corrosive wear, and surface hardness [4]. For instance, the prerequisite for pitting or crevice corrosion is surface roughness. The surface roughness effect often relates to the surface nucleation of metastable pit preceding the propagation of the pit. Furthermore, surface finishing is also believed to cause uneven corrosive wear when steels are in service. One of the main factors that often contribute significantly to corrosion is the degree of surface finishing of the material under investigation [2, 5].

In general, the initial stages of corrosion are highly dependent on the surface roughness because dense oxide films on metals and alloys surface protecting the metals/alloys from the corrosive medium can be easily damaged (e.g., during abrasion) at higher surface roughness [6]. Since many interaction factors may govern corrosion behavior, sometimes there are differences between experimental data and field data. One of the factors that might cause those differences is surface conditions. It was described theoretically that surface roughness could influence the electrochemical or mechano-chemical behavior of a surface. Such conditions could promote different interactions in the micro-electrical behavior and will impact the corrosion rate [2].

A number of studies have been carried out to investigate various aspects of surface roughness and modification in relation to the corrosion rate. Cheng and Roscoe [7] investigated the influence of surface polishing on the electrochemical behavior of

titanium. The researcher concluded at a high anodic potential range (>2.0 V), the 1 μm diamond paste polished electrode gave a much higher anodic current than the rough sandpaper polished. Klein et al. [8] investigated the erosion and corrosion resistance of chromium-nickel steel in the as-delivered condition, and the type of the heat-treated types of the steel has higher wear resistance.

Li et al. [9] investigated the significant effect of single and multiple steps pickling of 304 stainless steel in acid electrolyte. It was found that a smooth surface finishing was obtained with intergranular corrosion taking place for a single step pickling. However, multiple-step pickling in successive HCl greatly influenced surface finishing. Liu et al. [6] also examined the effect of CrN coatings produced by physical vapor deposition (PVD) technique on mild steel of different surface roughness. It was discovered that as the roughness of the substrate decreased, the number of flaws, evidenced by the frequency and size of initial corrosion sites, also decreased.

It is well recognized that the surface integrity of materials (machined components) has significant effects on their functional performance. Service failures related to corrosion almost always initiate from the surface or subsurface region. The corrosion resistance of steels and their correlation with surface integrity are now an important concern for product sustainability. In practice, corrosion can never be stopped but can be hindered to a reasonable level. Corrosion control is an important activity of technical, economic, and environmental importance. Hence, the objective of this study is to investigate the effect of surface preparation on the corrosion behavior of zinc-coated mild steel in seawater.

2. MATERIALS AND METHODS

2.1. Materials, Apparatus, and Equipment

The following materials were used in the course of this study: mild steel as substrate, Fischer zinc sprayed with grey color, acetone, seawater (corrosive medium), grinding file, emery papers, hydrochloric acid (HCl), sodium chloride (NaCl), grinding disc, distilled water, laboratory beaker, flask, plastic brush, and ethanol. Other apparatus and the equipment used are spark test machine (Spectomax LM806-Amatek, USA), hack saw, digital weighing balance, grinding and polishing machine, metallurgical optical microscope, and Digi-Ivy 2300 potentiostat.

The mild steel was obtained from Dorman Long Engineering Limited, Lagos, Nigeria. The chemical composition of the mild steel sample using Spectomax LM806-Amatek, USA, is presented in Table 1. The corrosive medium (seawater) was obtained from the Lagos lagoon front of the Atlantic Ocean and was analyzed in the chemistry laboratory of the University of Lagos. The composition of the seawater is presented in Table 2. The Fischer zinc spray FTC-ZS used was procured from a local vendor in Lagos, Nigeria. Fig. 1 shows the photograph of the Fischer zinc spray, and its composition is also presented in Table 3.

Table 1.

Chemical composition of the mild steel.

Elements	Fe	C	Si	Mn	Cr	Ni	w	Mo	Al
Wt %	98.582	0.099	0.137	0.770	0.024	0.026	0.236	0.061	0.066

Table 2. Composition of the seawater sample.

Parameters	Values
pH	8.67
Temperature	29.5°C
Conductivity	50,650 μ S/cm
Total dissolved solids	36,000 mg/L
Salinity	25,750 mg/L
Chloride	14,238.21 mg/L
Acidity	56 mg/L
Iron	0.45 mg/L

**Fig. 1.** Photograph of the Fischer zinc spray paint used.**Table 3.**

Chemical composition of Fischer zinc spray [10].

Ingredient	Concentration (%)
Propane	10 - 25
Zinc powder – Zinc dust	10 - 25
Kohlewasserstoffe, C9, Aromaten	2.5 - 10
Butane	2.5 - 10
Ethylbenzene	less than 2.5
Isobutane	10 - 25
Xylene	10 - 25
Acetone, propan-2-one, propanone	10 - 25

2.2. Preparation of Samples and Immersion Procedure

The as-received mild steel was machined into test samples of dimensions 20 mm x 30 mm x 4.5 mm. The test samples were designated for easy identification. The samples were surface modified using different techniques, as shown in Table 4. The samples were divided into two groups: sprayed and unsprayed samples for comparison, as presented in Table 4. They were washed in distilled water with a plastic brush and then soaked in ethanol prior to the experiment. This was done to ensure that all unwanted contaminants were properly removed and initial corrosion of the sample was prevented. Prior to the immersion of the samples, they were allowed to dry for 30 minutes. Some of the samples were then sprayed with Fischer zinc spray with the final coating taken as thick film putting into consideration the report of Fauchais and Vardelle [11]. The initial weight (W_i) of the sample was taken using a digital weighing scale. Thereafter, an equal proportion of the

analyzed corrosive medium of 300 ml was measured with a laboratory beaker and poured into transparent flasks. To achieve complete immersion of the samples, sewing threads attached to the ruler were used to suspend the samples in the corrosive medium (see Fig. 2). After every 7-days (168 hours) of immersion, the individual sample was brought out of the corrosive medium. They were gently and thoroughly cleaned with distilled water and ethanol and weighed for the final weight (W_f). The weight loss, $W_t = (W_i - W_f)$ obtained, was then used to calculate the corrosion rate. The dried samples weights were measured to the nearest 0.000g. In all cases, the processes of samples cleaning, washing, drying, and weighing were thoroughly and gently carried out – since the data collection largely depends on these processes, which is in accordance with the report of Ikechukwu and Ugochukwu [12]. To enhance the reliability of the results, each test was repeated twice. The experiments were monitored for a total of forty-two days.



Fig. 2. (a) Laboratory set-up and (b) potentiodynamic polarisation set-up.

Table 4. Samples preparation.

Test sample	Number of samples (coated and uncoated)	Surface preparation
P60	4	The sample was smoothened using emery papers of grit size P60
CC220	4	The sample was smoothened using emery papers of grit size P60, P80, and CC220, respectively
CC1200	4	Sample was smoothen using emery papers of grit size P60, P80, CC220, CC320, CC400, CC600, CC800, CC1200 respectively
Grinding disc	4	The sample was smoothened using a mechanical hand grinding machine. Using a fine grit of 80
Pickling	8	2 samples went through pickling in 3 mol of HCl while the other went through pickling in 3 mol NaOH Condition: at room temperature for 3 min each
Filing	4	Each surface of the sample was filed

2.3. Qualitative Analysis Method

In this work, a quantitative analysis technique for the determination of the corrosion rate based on the weight loss method was adopted. It involved taking the weight loss (Wt) of unsprayed and sprayed samples before and after immersion in the corrosive medium. The corrosion rate was calculated using Equations (1) and (2) in accordance with the report of Abel-Samad and Zaki [13].

$$\text{Corrosion rate, } C_t = \frac{87.6 \cdot \text{weight loss (W}_t\text{)}}{\text{Area (A)} \cdot \text{time (T)} \cdot \text{Density } (\rho)} \quad (1)$$

Where: Surface area(A) = 2[(L * B) + (L * H) + (B * H)] (2)

The weight loss was also used to calculate the surface coverage and inhibition efficiency using Equations (3) and (4) respectively after 1800 hours.

$$\text{Surface coverage, SC} = \frac{CR_a - CR_p}{CR_a} \quad (3)$$

$$\text{Inhibition Efficiency, IE (\%)} = 100 \text{ SC} \quad (4)$$

Where: CR_a and CR_p are corrosion rates in the absence and presence of the inhibitor, respectively.

2.4. Potentiodynamic Polarisation Technique and Microstructural Examination

A potentiodynamic polarization test was performed with cylindrical steel electrodes mounted in seawater. The steel electrode was prepared according to ASTM G59-97 [14]. The studies were performed at 25° C ambient temperature with Digi-Ivy 2300 potentiostat and electrode cell containing 200 ml of the acid media. Platinum was used as the counter electrode, and silver chloride electrode (Ag/AgCl) was employed as the reference electrode (see Fig. 2). Potentiodynamic measurement was performed from -1.5 V to +1.5 V at a scan rate of 0.0015 V/s according to ASTM G102- 89 [15]. The corrosion current density (I_{corr}) and corrosion potential (E_{corr}) were derived from the Tafel plots of potential versus log current. The corroded samples were examined with an optical microscope for surface defects. Prior to the microstructural examination, each of the samples was dried in an oven at 130° C for 5 minutes [16, 17].

3. RESULTS AND DISCUSSION

3.1. Corrosion Rate, Inhibition Efficiency, and Microstructure

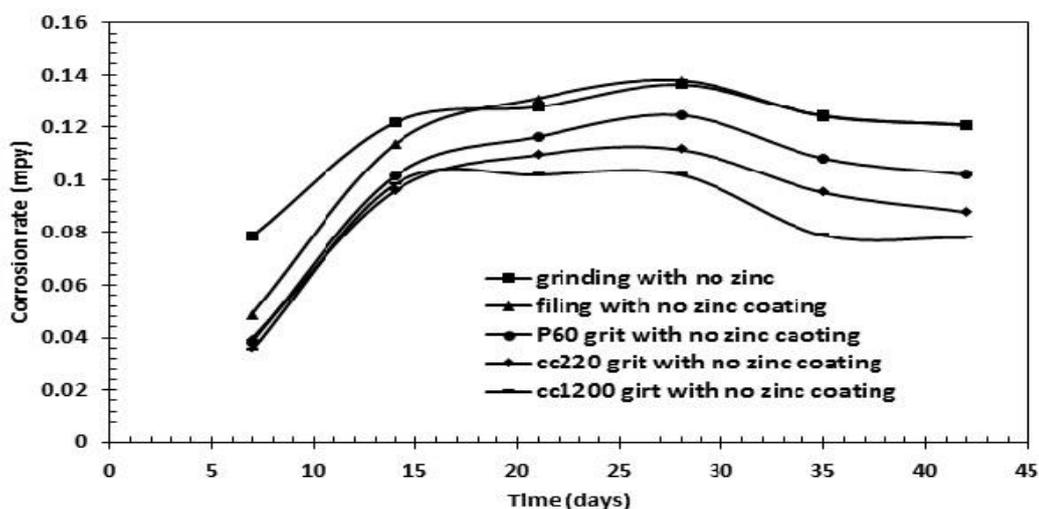
As shown in Table 4, different surfaces were prepared. The chemical analysis of the as-received Fisher zinc spray indicates that the zinc constituent (Table 3) that is often responsible for corrosion prevention is 10 - 25 % of the spray constituents. The chemical analysis of the corrosive medium is presented in Table 2. The salinity and chloride (mg/L) that were present in the corrosive medium are well above the required limit for normal water based on the report of W.H.O [18]. Thus, the seawater used is suitable for this study.

Fig. 3 shows the variation of the corrosion rate with the time of exposure for the mild steel sprayed and unsprayed with the zinc spray. In the unsprayed sample, three regions could be seen, and corrosion rate initially increased with exposure time, then it approached saturation and slightly decreased. These regions could be classified as the active, active-passive, and passive regions. Other samples of the different surface modifications exhibited a similar trend which indicates that the results are very consistent. The active-passive region commenced and finished at 14 and 28 days, respectively.

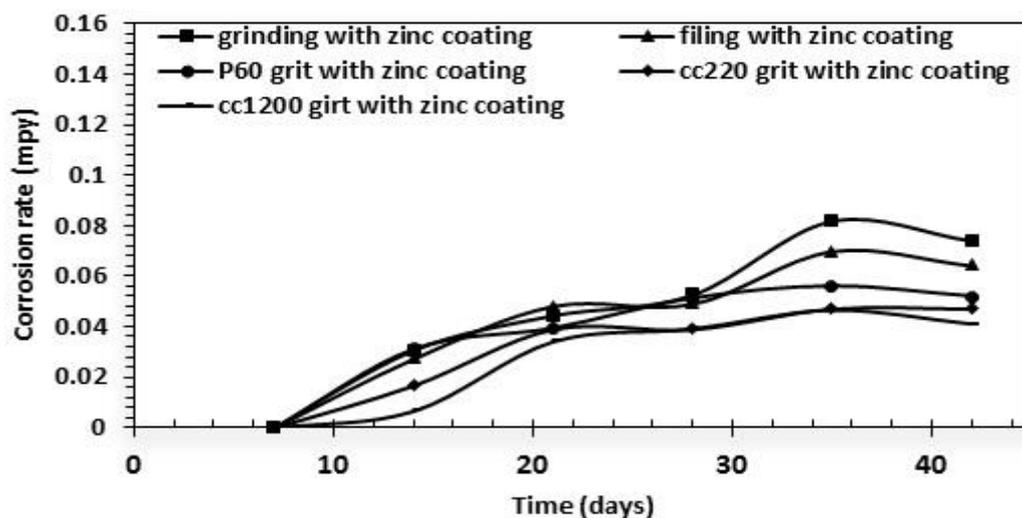
Fig. 3 of the unsprayed sample also indicates that CC1200 grit has the least corrosion rate closely followed by CC220 grit, then P60 grit, filing, and grinding. This can also be anticipated in Fig. 4, showing the photographs of the surface of the samples after immersion. The least corrosive effect was observed in the micrograph of the coated sample smoothed with CC1200 emery paper. For the coated/sprayed sample, the corrosion rate increased with exposure time with the predominately active region for 28 days, unlike the unsprayed sample that lasted for 14 days. No corrosion was observed in the first 7 days of the experiment independent of surface modification. The figure demonstrated that spraying

significantly reduced corrosion rate for the same exposure time. As the roughness of the substrate decreased, the number of contours, evidenced by the frequency and size of the initial corrosion sites, also decreased. The exposed area of the substrate is a function of the surface roughness – the higher the area exposed, the more the surface roughness [19]. With all measurements taken, corrosion was clearly reduced with surface modification and spraying. The low corrosion rate was due to the passivation of zinc [20]. The adhesion of spray on a rougher surface reduced the corrosion rate. However, the cracking of the adhered spray on the substrate was greatly influenced by the spray qualities, which include density, thickness, and porosity, which could be the cause of corrosion.

The initial aggressiveness of the uncoated samples immersed in seawater between 0 and 14 days can be attributed to surface modification of the substrate and corrosive medium used. The corrosion rate increased with increasing exposure time for each of the samples. For the same exposure time, the corrosion rate also increased due to the surface modification of the substrates. There was no barrier formed between the mild steel substrate and the corrosive medium, and more oxygen could be dissolved in the experimental setup. Thus, corrosion was very aggressive, and this agrees with the earlier report of Fauchais and Vardelle [11]. Beyond 14 days, the partial passivation experienced was due to saturation of dissolved oxygen since no spray was applied. This may also be due to the fact that corrosion products that often prevent further corrosion have been formed, which agrees with the report of Ayoola et al. [20]. The aggressiveness of corrosion rate is a function of the type of substrate, oxygen presence [11], corrosion products formed [20], and inhibitor presence. However, dissolved oxygen seems to be the most important, as earlier reported by Fauchais and Vardelle [11].

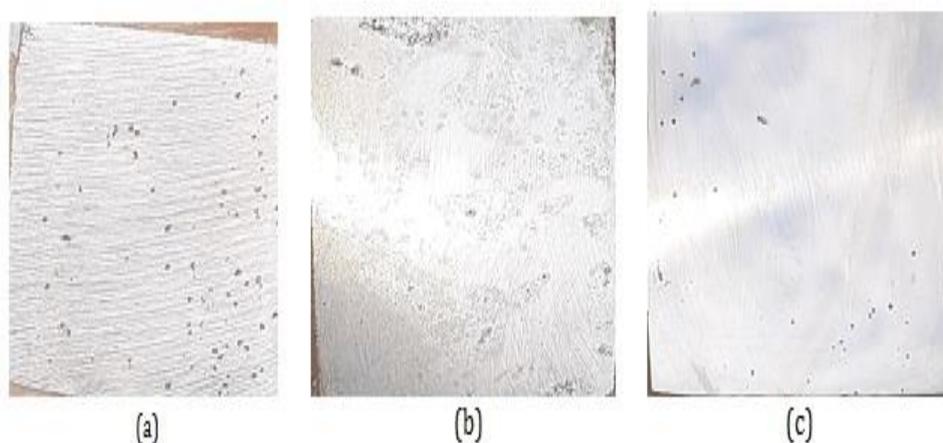


(a)



(b)

Fig. 3. Variation of corrosion rate with exposure time for (a) uncoated mild steel and (b) coated with different surface modifications.



(a)

(b)

(c)

Fig. 4. Photographs of the zinc-coated sample after potentiodynamic polarisation test (a) ground sample, (b) sample smoothen with CC220 emery paper, and (c) sample smoothen with CC1200 emery paper.

Fig. 5 shows the variation of corrosion rate with the exposure time when the samples were pickled in a sodium hydroxide (NaOH) and hydrochloric acid (HCl) solutions. The samples went through pickling in 3 mol of NaOH and HCl solutions and thereafter sprayed with zinc. Two regions could be identified. The corrosion rate was enhanced by the exposure time and higher with HCl compared to NaOH (region 1). The graphs seem to stabilize just beyond 28-days, with the commencement of active-passive area (region II). In region, I, the reaction of the corrosive medium (electrolyte) with mild steel was prompted by cracks and micro-channels that were readily available within the zinc spray through the oxide scales and towards the modified surfaces. The delayed migration of the corrosive medium

prompted no corrosion in the first 7-days. On wetting the surfaces of the mild steel, the corrosion rate depended on the dissolved oxygen used in the pickling solution. The dissolved oxygen content, however, decreased from the outer oxide layer to the inner oxide layer. The corrosion rate of the sample in sodium hydroxide (NaOH) solution is lower than the corrosion rate of the mild steel sample in hydrochloric acid (HCl) solution due to passivation because of the formation of a protective oxide/hydroxide layer (film) [21] which inhibited corrosion. It has been reported that in alkaline media, the film corresponds to a double-layer consisting of inner magnetite and an outer ferric oxide according to a $\text{Fe}_3\text{O}_4/\text{Fe}^{3+}$ structure [22, 23].

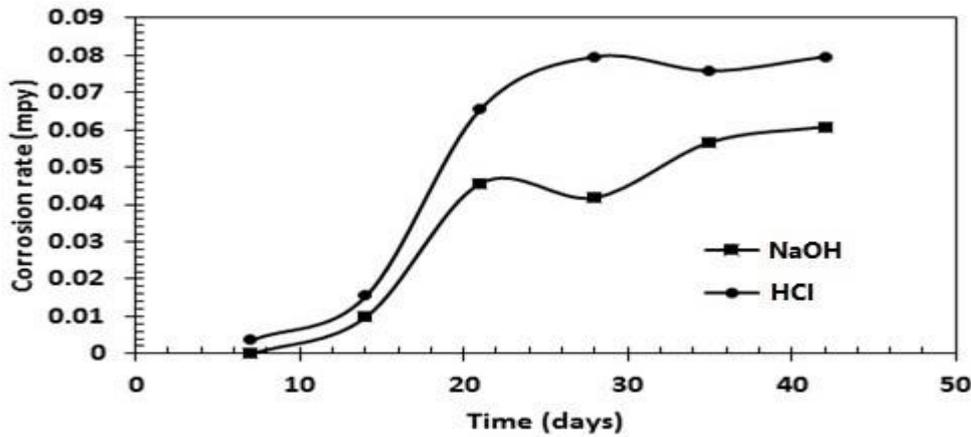


Fig. 5. Variation of corrosion rate with exposure time when samples were pickled in NaOH and HCl solutions.

The dependence of the inhibition efficiency on the exposure time for different surface preparation investigated is shown in Fig. 6. Surface coverage (SC) shown in “Eq. (3)” was used to obtain the inhibition efficiency using Equation 4. The inhibition efficiency decreased with increasing exposure time. The inhibition efficiency of 100 % was achieved for the first-seven days independent of the surface preparation due to the presence of zinc spray. This is

because there was no effect of corrosion on the substrate for the first seven days. Similar inhibition efficiency can also be seen on 21 and 28 days. On the 14 and 35 days, the inhibition efficiency was 75 and 90 %, respectively. This suggests that 100 % inhibition efficiency is possible when adequate zinc spray is deposited independently on the surface preparation.

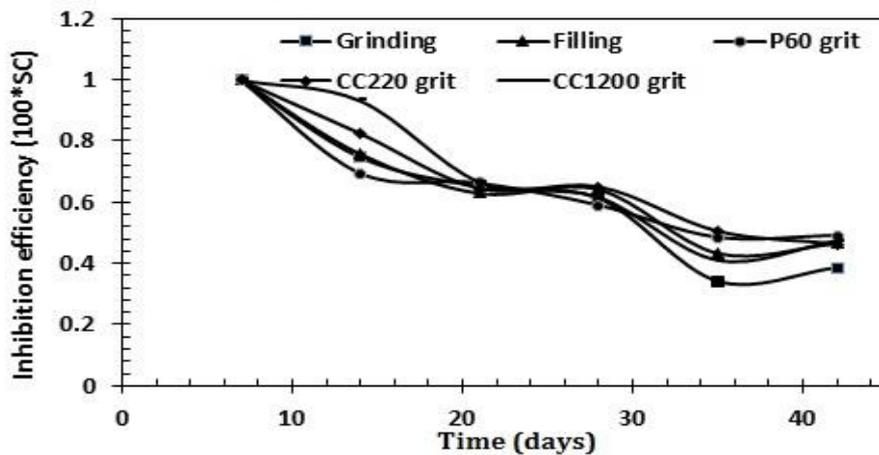


Fig. 6. Dependence of inhibition efficiency on the exposure time for different surface preparation.

3.2. Potentiodynamic Polarisation

Fig. 7 shows the potentiodynamic anodic polarization curves of the mild steel immersed in the corrosive medium for all surface modification with and without zinc spray. Fig. 8 depicts pickling surfaces with and without zinc spray. The comparison of samples ground and filled with and without zinc spray extracted from Fig. 8 is shown in Fig. 9. Obviously, the corrosion potential from the figure tends towards the positive direction for the samples painted. The corrosion current also tends towards the lower values for the painted samples compared to the samples without spraying. This indicates the inhibiting effect of zinc paint. However, the analysis of the corrosion current indicates that sprayed samples exhibited better corrosion resistance due to lower values of corrosion current.

Presented in Table 5 are the detailed electrochemical corrosion parameters derived from the potentiodynamic polarization curves for all the samples investigated. In this table, a comparison of the corrosion potential and corrosion density for different surface preparation (with and without zinc spray) indicates that there is an increase in corrosion potential and a decrease in corrosion current. This behavior is not different when the samples were pickled in NaOH and HCl prior to painting. The sample pickled in NaOH exhibited higher inhibition efficiency than that of HCl, which further confirmed the result of Fig. 6. However, the effect of painting seemed less important when the samples were immersed in HCl and NaOH solutions as the corrosion potential, and current remained the same. Apparently, spraying and surface preparation played a significant role in corrosion rate and its inhibition

efficiency on the samples. The possible reason could be due to the fact that zinc spray caused the formation of an amorphous phase within the matrix of the corrosive medium, thereby enhancing the corrosion resistance property of the mild steel

compared to samples with no zinc spray. However, the performance of the zinc spray diminished over time due to depreciation in the formed amorphous phase, which agrees with the report of Ayoola et al. [20].

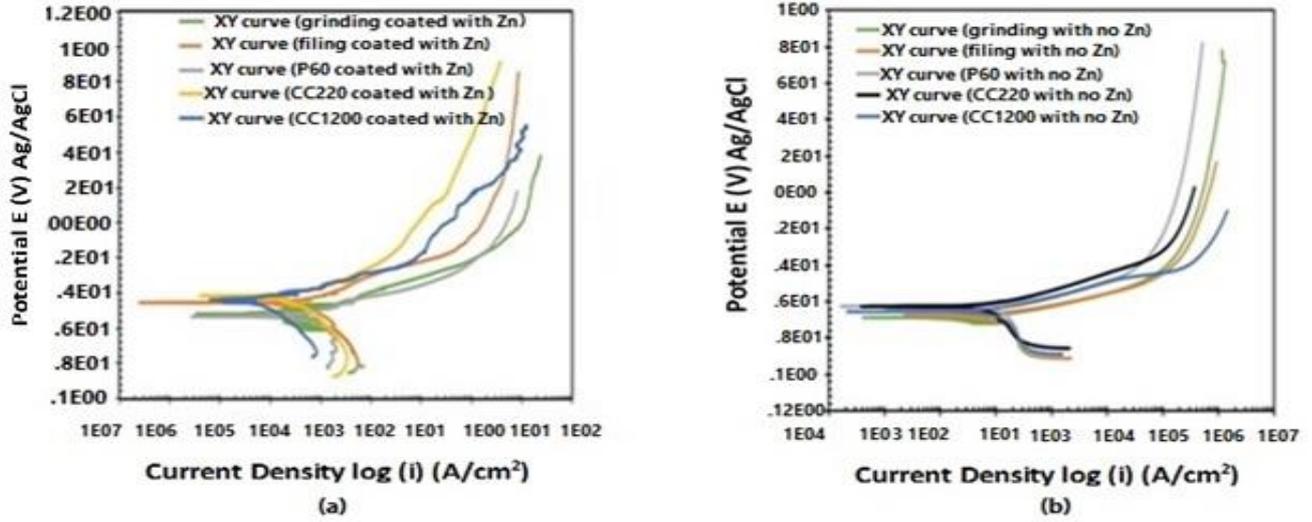


Fig. 7. Potentiodynamic anodic polarization plots of mild steel immersed in seawater (a) with Zn spray (b) without Zn spray.

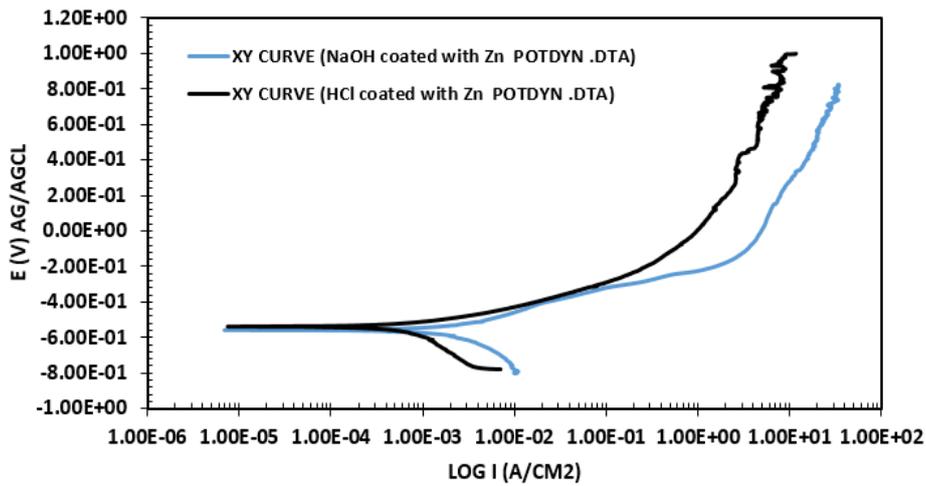


Fig. 8. Potentiodynamic anodic polarisation plots of mild steel immersed in seawater with pickling treatment and Zn spray.

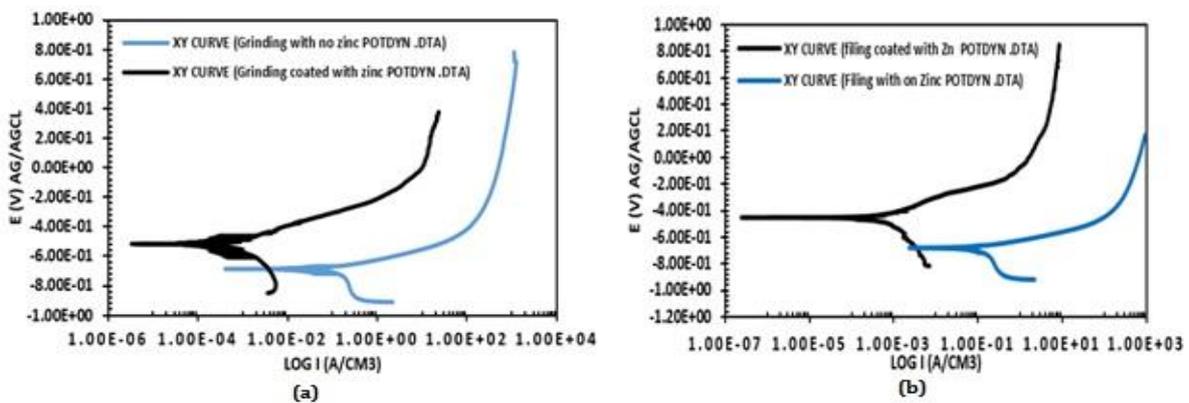


Fig. 9. Potentiodynamic anodic polarisation plots of mild steel immersed in seawater ground and (a) filled surfaces with Zn spray (b) without Zn spray.

Table 5.

Electrochemical corrosion parameters of mild steel immersed in corrosive medium surface preparation for sprayed and unsprayed samples.

Surface	Sprayed samples		Unsprayed samples	
	Corrosion potential (E_{corr}) (V)	Corrosion current density (I_{corr}) (A/cm ²)	Corrosion potential (E_{corr}) (V)	Corrosion current density (I_{corr}) (A/cm ²)
Grinding	-4.90*10 ⁻⁰¹	3.49*10 ⁻⁰³	-7.29*10 ⁻⁰¹	1.57*10 ⁻⁰¹
Filing	-4.95*10 ⁻⁰¹	7.11*10 ⁻⁰⁴	-7.11*10 ⁻⁰¹	1.29*10 ⁻⁰¹
P60 Grit	-5.62*10 ⁻⁰¹	7.03*10 ⁻⁰⁴	-6.12*10 ⁻⁰¹	1.26*10 ⁻⁰¹
CC220 Grit	-3.89*10 ⁻⁰¹	3.78*10 ⁻⁰⁴	-6.03*10 ⁻⁰¹	1.03*10 ⁻⁰¹
CC1200 Girt	-3.83*10 ⁻⁰¹	1.01*10 ⁻⁰⁴	-5.85*10 ⁻⁰¹	1.07*10 ⁻⁰²
HCl	-5.78*10 ⁻⁰¹	7.40*10 ⁻⁰³	-3.28*10 ⁻⁰¹	5.40*10 ⁻⁰¹
NaOH	-4.86*10 ⁻⁰¹	2.62*10 ⁻⁰³	-4.86*10 ⁻⁰¹	2.62*10 ⁻⁰²

The corrosion rate (C_r) is given in Equation (5). The density of the mild steel substrate is 7.85 g/cm³ and the equivalent weight of the corroding metal. For this condition, the corrosion rate is proportional to the corrosion current density (I_{corr}) and an equivalent weight of the corroding metal [24].

$$C_r = \frac{0.00327 * \text{Corrosion current density } (I_{corr}) * \text{equivalent weight of the corroding metal } (E_q)}{\text{Density } (D)} \quad (5)$$

3.3. Surface Preparation

The effect of surface preparation on the corrosion resistance of the samples is clearly observed in Fig. 10. The sample whose surface was smoothed using emery papers of grit size CC1200 and coated with Zn spray exhibited the least corrosion rate of 1.35×10^{-9} mpy when compared with others. This implies that it has the highest corrosion resistance. This shows that surface roughness also influenced the rate of corrosion. The rougher the surface, the higher the corrosion rate or, the smoother the surface, the lower the corrosion rate.

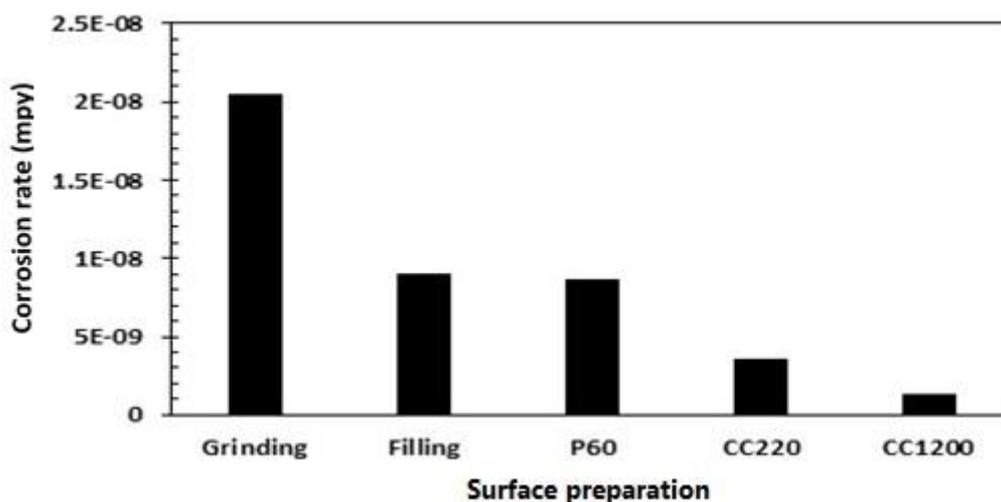


Fig. 10. Corrosion rate as a function of surface preparation.

4. CONCLUSIONS

The main conclusions of the present study can be summarized as follows:

Surface preparation significantly affected the corrosion rate of mild steel – the rougher the surface, the higher the corrosion rate. The surface preparation has no significant effect on the mild steel in the first seven days of immersion due to the presence of zinc spray. The active-passive region of the immersed mild steel is a function of the exposure time, surface preparation, and inhibitor used. The results also indicated that the corrosion rate of the test sample was higher in hydrochloric acid solution (HCl) than sodium hydroxide (NaOH) solution. The inhibition efficiency of the Fisher zinc spray used to range between 75 and 100 %. Both corrosion potential and corrosion current were affected by the

surface preparation of the mild steel. The sample prepared with CC1200 grit paper and further coated with zinc spray exhibited the lowest corrosion rate of 1.35×10^{-9} mpy indicating the efficacy of coating.

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