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EMPLOYING *Cymbopogon citratus* (LEMONGRASS) AS ECO-FRIENDLY CORROSION INHIBITOR FOR MILD STEEL IN SEAWATER

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Abstract: *Cymbopogon citratus* (lemongrass) is a common plant in Southeast Asia that is rich in phytochemical compounds, such as flavonoids, phenolics and tannins. These compounds play an essential role in corrosion-inhibition processes. The characterization of the extract was done by Fourier Transform Infrared and UV-Vis Spectroscopy. The anticorrosion action of lemongrass extract was evaluated for mild steel through immersion in seawater medium using weight-loss and Electrochemical Impedence Spectroscopy. The surface analysis was carried out by optical and Scanning Electron Microscope. The mild steel grade JR235 was prepared in size of 25 mm × 25 mm × 3 mm and was polished using a stand grinder. The immersion test was conducted in seawater with different concentrations of lemongrass extract for a duration of 50 days. Meanwhile, the amount of lemongrass extract incorporated varied from 5%, 10%, 15% to 20%. Six characterizations were conducted in this study which are weight-loss study, Fourier Transform Infrared, UV-Vis spectroscopy, Electrochemical Impedance Spectroscopy, Optical Microscope and Scanning Electron Microscope. In this study, a major constituent recorded was lawsone, which can impede corrosion on metal surfaces. The results indicated that weight loss without additive was higher compared to other samples with additives. The weight-loss results show that 10% and 20% additive managed to reduce corrosion by 0.07104% and 0.16234% compared to without additives, respectively. Charge transfer resistance increases up to $3.2 \times 10^6\ \Omega.cm^2$ with the presence of 20% additive. Surface studies results reveal that additives significantly improve the smoothness of the mild steel surface. Thus, this study finds that lemongrass is a potential functional natural additive for steel, particularly mild steel in seawater medium..

Keywords: *Corrosion, Electrochemical Impedance Spectroscopy (EIS), FTIR Spectrum, Inhibitor, Lemongrass Extract*.

Introduction

Corrosion of mild steel when in contact with circulating water is a common problem in the marine industry. The corrosive nature of circulating water comes from dissolved oxygen and the concentration of aggressive ions, such as chloride and sulphate, can accelerate marine corrosion (Saremi *et al*., 2006).

For the good of humans and the environment, corrosion additives that are environmentally friendly ought to be produced (Abdel-Gaber *et al*., 2006; Deyab *et al*., 2017). Green additives which comprise non-toxic compounds to humans have low impact on the environment, are biodegradable, efficient and cost-effective (El Bribri *et al*., 2013; Ibrahim *et al*., 2016; Prabakaran *et al*., 2016). Extracts of plant parts, such as leaves, peels, seeds, fruits and roots, have been reported as effective corrosion additives for metals in different aggressive conditions (Ji *et al*., 2015; Ibrahim *et al*., 2016; Chaubey *et al*., 2017). The use of natural products extracted from plants as a corrosion additive has become a key area of research because they are extremely rich sources of naturally synthesized chemical compounds that are biodegradable, renewable, cost-effective and can potentially be extracted using simple methods (Ramde *et al*., 2014; Saeed *et al*., 2019; Umoren *et al*., 2019).

Cymbopogon citratus (lemongrass) is an aromatic plant belonging to the Gramineae family. It is a tall, clumped, perennial grass growing to a height of 1 m. The leaf-blade is linear, tapered at both ends and can grow to a length of 50 cm and a width of 1.5 cm (Samy *et al*., 2005). The leaf-sheath is tubular in shape and acts as a pseudostem. This plant produces flowers at matured stages of growth. Lemongrass is also an aromatic plant that is used in traditional foods due to its lemon-like flavour and it is also employed in popular medicine (Deyab *et al.,* 2017). Lemongrass is a local herb that is convenient to obtain and reasonably cheap. Lemongrass is rich in phytochemical compounds such as flavonoids, phenolics and tannins. These three elements combine with the metal cations and adsorb on the metal surface to protect metallic parts from corrosion. Lemongrass has properties that meet requirements to be used as a future corrosion additive.

Since lemongrass has a vast potential to be used in marine applications, research has been conducted to test the possibility of using it in preventing corrosion on mild steel. Besides, it possesses environmentally friendly properties and is easily accessible. It could be used to paint the ship hull that is exposed to salt water or moisture all the time. Hence, it can be said that it has the ability to minimize the corrosion rate and extend the ship hull lifespan. Corrosion inhibition performance of lemongrass on mild steel in a seawater environment was studied by real-time tests using corrosion rate, electrochemical and weight-loss methods.

Materials and Methods

Materials

The metal used was 75 plates of mild steel, grade JR235, with dimensions of 25 mm x 25 mm x 3 mm. A 2 mm hole was drilled through the metal by using a PF-DS-d system PaoFong PF-2VS milling machine at a constant speed of 600 rpm in order to hang the metal in seawater medium. The steel coupons were polished with a series of emery papers, from grade 320 to grade 600, with the help of a BOSCH GWS24 polisher. The coupons were then degreased with acetone, followed by washing with distilled water. The coupons were allowed to dry, at room temperature of 30^{o}C (+/-1^{o}C). The coupons were then stored in a desiccator to keep them away from moisture before the coating process.

Preparation of Lemon Grass Extract

The extraction was carried out by placing 150 g of dry lemongrass (taken from sliced lemongrass sample) in a 1 liter flask and 500 mL of N-hexane solvent. The flask and content mixture were allowed to sit for 36 hours for the complete extraction of oil from the lemongrass. After which, the extract was decanted into a 1 liter beaker and the remaining plant matter was further extracted with 200 ml of ethanol to dissolve some other ethanol-soluble essential oils. The mixture was then transferred to a 500 ml separating funnel and separated by liquid-liquid extraction process. The contents of the separating funnel were allowed to come to equilibrium, which separated into two layers (depending on their different densities). The lower ethanol extract and the upper hexane layer were collected into two separate 250 ml beakers before placed in a water bath at 78°C.

Apparatus Setup

The dried metal that has been cleaned and stored in the desiccator was prepared for coating. Each metal was coated by immersion method. One side of the metal was immersed in a 750 ml container containing a sample mixture that has been prepared. The coated metal panels were allowed to dry at room temperature. When it was visually dry, the same step was repeated to coat the other side of the metal. The process of immersing and drying was repeated 2-4 times in order to get a constant coat thickness. The coat thickness was measured by a thickness gauge Positector 6000 and the thickness obtained was approximately 110 +/- 10 microns which was sufficient for a protective layer according to ASTM E376-17 standards. All the metals that were fully coated and dried were then stored in the desiccator before subject to immersion tests.

An aquarium 30 x 30 x 60 cm in dimensions was prepared for the immersion test with seawater. Next, the metal plates that were coated with different percentages of lemongrass extract (later will be known also as an inhibitor) such as 0%, 5%, 10%, 15% and 20%, had their weights recorded before being tied and hung with 8 lbs nylon string for 50 days. The metal specimens were taken out every 10 days to record their weight, and subject them to electrochemical tests, corrosion rate analysis and inhibition efficiency calculation.

Model information for other testing and analytical equipment used in this research are shown in Table 1.

Results and Discussion

Fourier Transform Infrared Spectroscopy (FTIR) of Lemongrass Extract

Fourier Transform Infrared Spectroscopy (FTIR) was the method used to obtain an infrared spectrum of absorption, emission, photoconductivity, or Raman scattering, of a solid, liquid or gas. The FTIR spectrum of the inhibitor was used to determine the functional groups based on the additive's characteristic vibration. In this study, the Thermo Nicolet 380 FTIR spectrometer was used.

The FTIR spectrum of lemongrass extract (Figure 1) shows two important frequency regions of the band, which are hydroxyl (OH) and carbonyl (C=O). A broad and strong peak around 3412.55 cm^{-1} shows the stretching vibration of the hydroxyl group (OH). The adsorption bands which appeared at 1697.16 cm^{-1} were attributed to carbonyl (C=O) while the C=C functional group was found at 1638.67 cm^{-1}. Also, peaks 1240.45 show the presence of (C-N) stretching and 1364.42 shows (OH) bending. The presence of a double carbon bond in the functional group indicates good corrosion resistance (Hajar *et al*., 2017). The phenol group of lawsone would donate its electron to the metal in order to achieve its noble state orbital, while the metal would gain the electron to become more stable. Moreover, this process indirectly minimizes further redox reaction and could resist metal from corrosion attack (Wan Nik *et al*., 2011).

It can be concluded that, with the presence of functional group peaks, one of the bioactive compounds responsible for inhibiting corrosion is the Citral compound, which is shown in Figure 2. As the percentage of Citral was about 70-80%, this proves that it is the major compound in lemongrass. The most efficient adsorption of the corrosion inhibitors requires the occurrence of heterocyclic structures containing heteroatoms N, S, O and/or P, double bonds (-C=C-), and lone-pair electrons (Qiang *et al*., 2017; Adam *et al.,* 2018). The spectrum shows the presence of (C=O), (C=C) functional groups, which are also in the structure of the Citral compound. Generally, organic inhibitors have heteroatoms, as shown in the Citral structure, which helps in inhibiting the corrosion process.

Table 1: Model for each testing equipment

Testing and analytical equipment	Brand and Model
Fourier Transform Infrared (FTIR) spectrometer	Thermo Nicolet 380
Ultraviolet-visible (UV-Vis) spectroscopy	Shimadzu 1800
Scanning Electron Microscope (SEM)	JEOL JSM-6390LA
Electrochemical Impedance Spectroscopy (EIS) machine	PGSTAT 302N

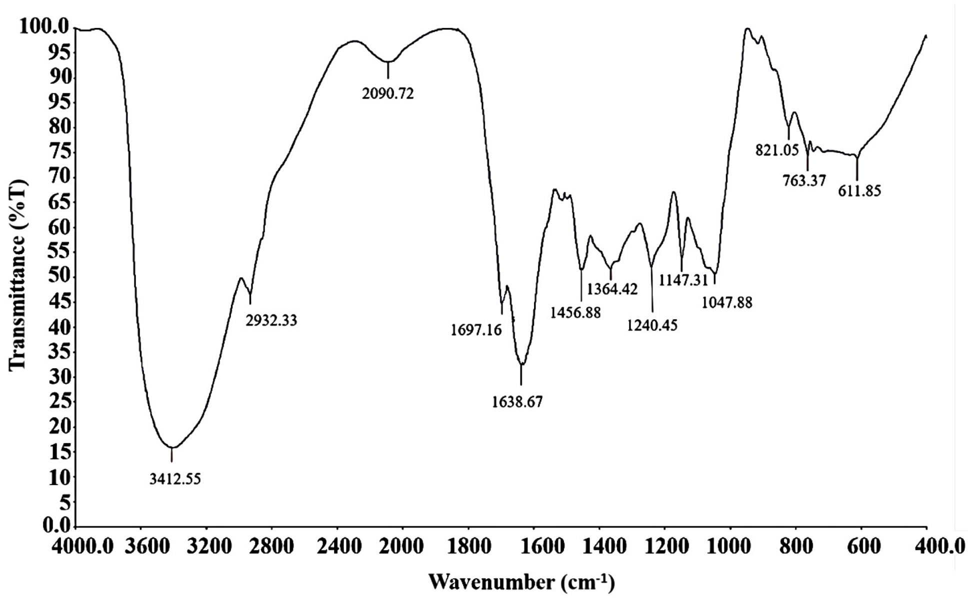

Figure 1: IR spectra of lemongrass extract

The antioxidant properties of Citral because it contains heteroatoms also can help in preventing corrosion. Furthermore, Citral contains polar functions with oxygen atom in the conjugated system, which have reported to exhibit good inhibiting properties. Oxygen is one of the active centres for the adsorption process on the metal surface because of the higher basicity and electrons. Corrosion inhibitors function mostly by modifying the surface of the metal via adsorption of the inhibitor molecules resulting in the formation of a protecting layer (Umoren *et al.,* 2018). The resulting adsorbed layer of extract acts as a barrier that separates the metal from the corrodent, therefore, the corrosion of the metal can be prevented. Thus, the use of an organic inhibitor is one of the most practical methods for the protection of the metal surface against corrosion (Singh *et al.,* 2018).

Fourier Transform Infrared Spectroscopy (FTIR) is a reliable method to study the adsorption of a layer. From the IR spectrum (Figure 1), it can be inferred that these are from one or two bio-active compounds and this may be the spectrum of hexanoic acid. If these compounds are hexanoic acids, then the analysis is as follows: the carbonyl stretch C=O of a carboxylic acid appears as an intense band from 1760-1690 cm^{-1}. The exact position of this broad band depends on whether the carboxylic acid is saturated or unsaturated, dimerized or has internal hydrogen bonding.

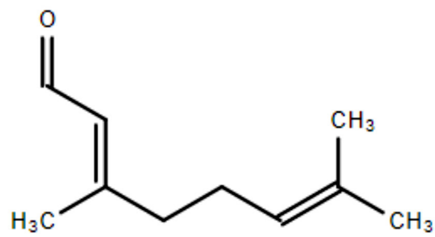

Figure 2: Citral structure

- O–H stretch from 3300-2500 cm^{-1}
- C=O stretch from 1760-1690 cm^{-1}
- C–O stretch from 1320-1210 cm^{-1}
- O–H bend from 1440-1395 and 950-910 cm^{-1}

The different vibrations of the different functional groups in the molecule give rise to bands of differing intensity. This is because $\partial\mu/\partial x$ is different for each of these vibrations. For example, the most intense band in the spectrum of hexanoic acid shown in Figure 1 is at 3412, 2932 cm^{-1} and is due to stretching of the C-OH bond. One of the weaker bands in the spectrum of hexanoic acid is at 1638 cm^{-1}, and it is due to long-chain methyl rock of the carbon-carbon bonds in octane. The change in dipole moment with respect to distance for the C-H stretching is greater than that for the C-C rock vibration, which is why the C-H stretching band is more intense than C-C rock vibration.

The assumed compound is as follows:

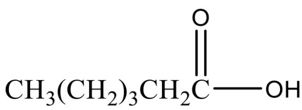

Figure 3: Hexanoic Acid.

Ultra-Visible (UV-Vis) Spectroscopy

From the IR Spectrum analysis, we assumed that one of the bioactive compounds to inhibit corrosion is Citral. The analysis of this compound also can be detected by using UV-Vis Spectrophotometer to determine the wavelength and absorbance of the compound. Based on the spectrum obtained in Figure 4, the λ_{max} is shown at 273.50 nm at absorbance 0.850, which is the highest peak. According to Miron *et al.* 2014, they obtained 270 nm for λ_{max} of the Citral compound. From this prior study, we conclude that the λ_{max}, or the first peak on the spectrum, was the Citral compound as it is the major compound in lemongrass. Next, the absorption was used for the calibration curve to determine the concentration of the phenolic compound and flavonoid.

From the IR Spectrum analysis, we assumed that this bio-active compound is hexanoic acid. Organic acids are known to determine the taste and flavor of foods. In addition to the food sector, many other areas, such as the pharmaceutical and chemical industries, environmental analysis and biotechnology, analyze organic acids. Analysis methods of organic acids include UV detection (detection of absorption of carboxyl groups), electrical conductivity detection (detection of ionic compounds), using pH indicators (visible absorption detection of pH changes caused by acidic components) and a post-column derivatization for high selectivity of detection. An appropriate method must be selected based on the analysis samples and for what purpose.

From the five peaks, it can be inferred that the first peak is probably a hexanoic compound and it will be the maximum amount from the extract. The second peak is not very sharp, meaning that it may be two or more unclear compounds. The third and fourth peaks indicate some components exist but ignorable. The fifth peak clearly shows a component but at a very low concentration in the extract.

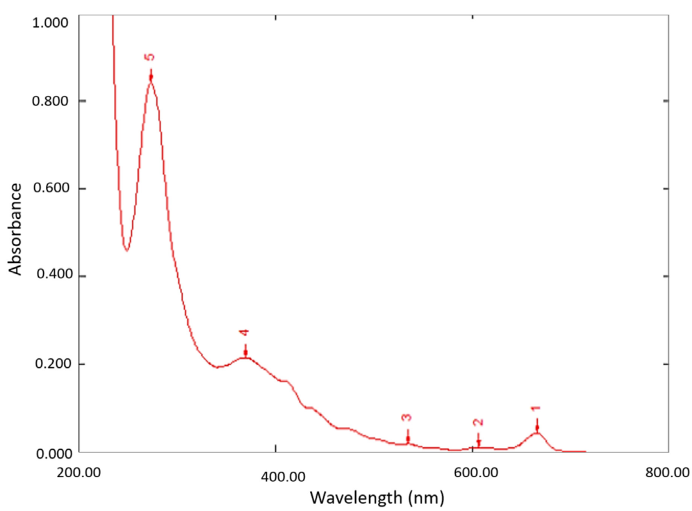

Figure 4: Ultraviolet-Visible (UV-Vis) spectrum of lemongrass extract

Refractive and absorption indices in the UV and visible region of selected aqueous organic acids and relevant hexanoic acids are reported. The acids investigated are aliphatic dicarboxylic acids oxalic, malonic, tartronic, succinic and glutaric acid. We also report data for pyruvic, pinonic, benzoic and phthalic acid. To cover a wide range of conditions, we have investigated the aqueous organic acids at different concentrations spanning from highly diluted samples to concentrations close to saturation.

The density of the investigated samples is reported and a parameterisation of the absorption and refractive index that allow the calculation of the optical constants of mixed aqueous organic acids at different concentrations is presented. The single scattering albedo was calculated for two size distributions using measured and a synthetic set of optical constants. The results show that hexanoic acid consisting of only these organic acids and water, have a pure scattering effect.

Electrochemical Impedance Spectroscopy (EIS)

EIS measurements were performed on the coated metal coupons in the presence and absence of lemongrass extract. Figure 5 shows the impedance plot showing a single capacitive semicircle indicating that the process was controlled by charge transfer and could be assigned to the mild steel oxide layer. Table 2 presents the data on the impedance gain via the EIS. The EIS study was conducted to compare the charge transfer resistance (R_{ct}) and double layer capacitance (C_{dt}) values of the protective layer in the presence and absence of lemongrass extract. Higher charging resistance (R_{ct}) could be attributed to higher inhibitor efficiency (Wan Nik *et al*., 2012; Noreen *et al*., 2017). Twenty percent of the inhibitor is the best concentration as it shows the highest charge resistance (R_{ct}) value of 3.2224 x 10^6 Ω.cm^2 and lowest value of double-layer capacitance (C_{dl}) that is 0.2712 µF/cm^2. The increment of charge transfer (R_{ct}) could be attributed to a lower capacitance of the double layer (C_{dl}). Thus, when the value of the charge transfer resistance (R_{ct}) increases, the double layer resistance (C_{dl}) decreases slightly with the increase of the concentration, which consequently causes the rate of corrosion to decrease.

The resistance of transmission charges (R_{ct}) for the other concentration continues to increase from the control to the best percentage of lemongrass extract, which was 20%. In contrast, double layer capacitance (C_{dt}) continues to decrease from control to the best lemongrass extract concentration of 20%. The equivalent electrical circuit is shown in Figure 6, where it shows three elements known as solution resistance (R_s), charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}).

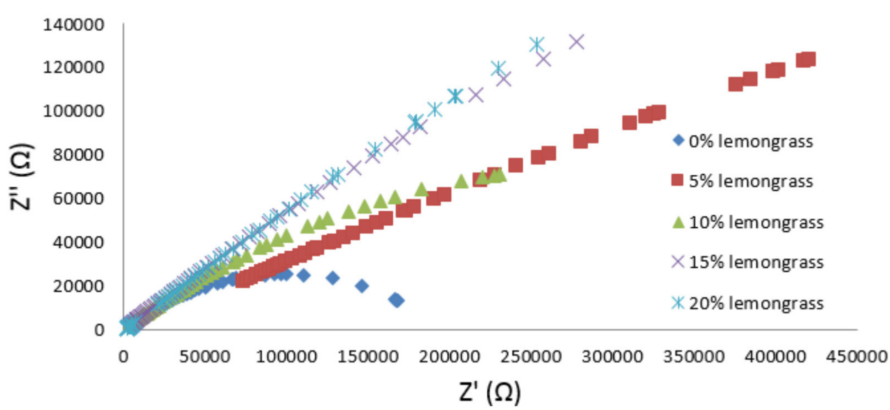

Figure 5: Nyquist plot for day 50

Table 2: Electrochemical parameters of coated mild steel in the presence and absence of lemongrass extract

Concentration (%)	**Charge Transfer Resistance, R_{ct} (Ω.cm²)**	**Double Layer Capacitance C_{dl} (μF/cm²)**	**CPE.N**
Control	0.1964×10^6	8.9830	0.98845
5	0.5867×10^6	8.1329	0.98838
10	1.7717×10^6	5.3596	0.98815
15	2.9695×10^6	1.0751	0.98803
20	3.2224×10^6	0.2711	0.98627

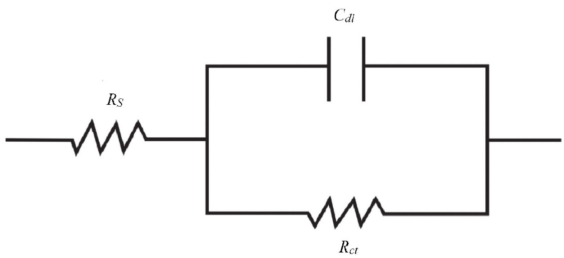

Figure 6: The fitted equivalent circuit for the samples in the absence and presence of lemongrass extract

Weight-Loss Measurement

Weight-loss calculation was one of the methods to determine the corrosion rate. The weight of mild steel before being immersed in corrosive media and the weight after immersion were recorded. By taking into account the surface area, density and time of immersion, the corrosion rate was calculated. When minimum corrosion occurs, the weight loss percentage is small. The data obtained from the weight loss calculation is tabulated in Table 3. Figure 7 illustrates weight-loss percentage through a graphical method for immersion period of 10, 20, 30, 40 and 50 days, with 5% of error. The thickness of the coated mild steel influences the value of the weight loss between all the samples.

As the concentration of lemongrass extract decreases, the weight-loss rate increases, indicating a higher rate of corrosion. Figure 7 shows a large weight loss of coated mild steel without the additive during immersion of 50 days, followed by mild steel with a concentration of lemongrass of 5%, 10%, 15% and 20%. It shows that an increase in the concentration of lemongrass extract in the protective layer increased the resistance to corrosion, which indirectly reduced mild steel weight loss.

Table 3: Weight loss percentage and inhibition efficiency of coated mild steel for day 10 and 50

Concentration (%)	**Immersion Period (days)**			
	10		**50**	
	Weight loss (%)	**Inhibition Efficiency (%)**	**Weight loss (%)**	**Inhibition Efficiency (%)**
Control	0.37057		0.48439	
5	0.32161	13	0.45598	6
10	0.33288	10	0.41335	15
15	0.30510	18	0.38332	21
20	0.27544	26	0.32205	34

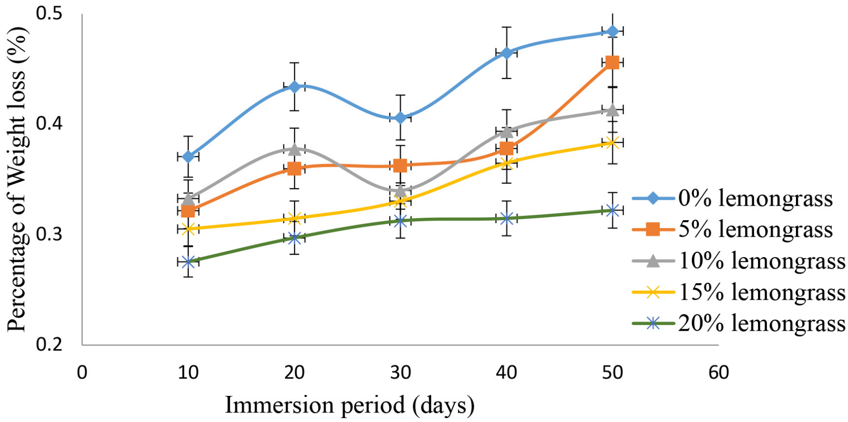

Figure 7: Graph of weight loss (%) against the immersion period (days)

Microscope Optic Result

Surface analysis was conducted on the mild steel surface by using an optical microscope for each lemongrass concentration until day 40. This analysis was conducted in order to fulfil the last objective for this study, which is to study the morphology and surface area of mild steel with optical microscope and SEM. Generally, there were changes in colour for each specimen, with the brown colour indicating rusting, as shown in the circled area in Figure 8. On day 20, more corrosion was observed on the control specimen compared to the mild steel coated with lemongrass extract concentrations of 5% and 10%. Mild steel coated with concentrations of 15% and 20% show few forms of corrosion. On day 30, the corrosion form increased, and mild steel coated with 15% concentration of lemongrass extract shows the rusting product. On day 40, corrosion was attacking the mild steel surface vigorously. As the immersion period increased, the mild steel coated with 20% concentration of lemongrass extract also showed changes that indicate the formation of corrosion. The colour for each specimen was more brownish.

Meanwhile, there were errors while conducting the experiment that can affect the

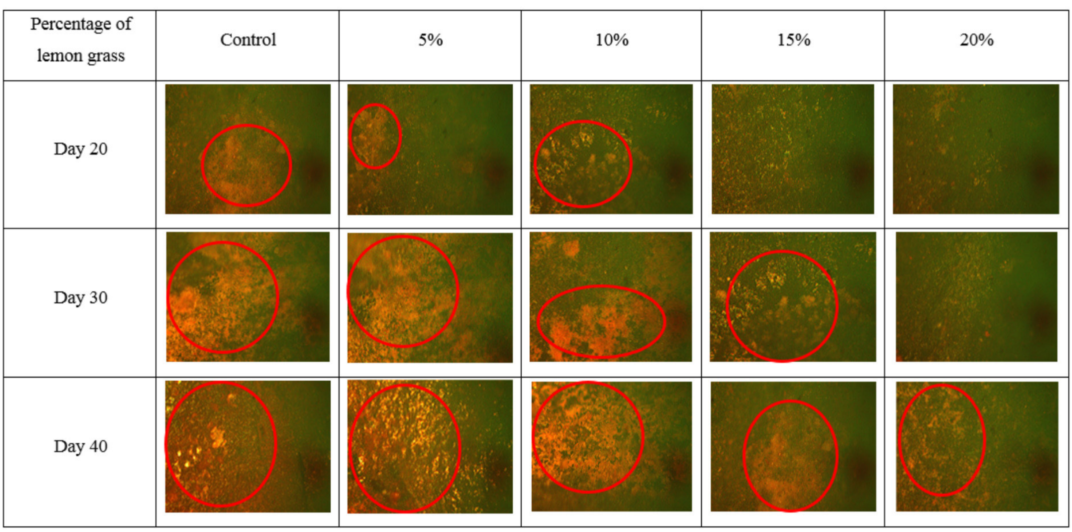

Figure 8: Image of mild steel surface after immersion test by using a microscope optic

corrosion process, such as the specimen was placed in very harsh conditions, unsuitable protective layer thickness that was caused by the uneven thickness of the protective layer by using dip coating method. These errors need to be rectified in further studies.

Scanning Electron Microscope

The scanning electron microscope imagery on the surface of mild steel type JR235 during immersion test for 50 days, in the absence and presence of lemongrass extract, are shown in Figure 9.

The initial specimens as shown in Figure 9 (a) exhibits relatively smooth surface morphology with small, fine scratches resulting from grinding and polishing on the surface of mild steel. The surface was also flat, clean, non-porous and there are almost no holes. While Figure 9 (b) shows corrosion product formed on the surface morphology of coated mild steel without additives after 10 days

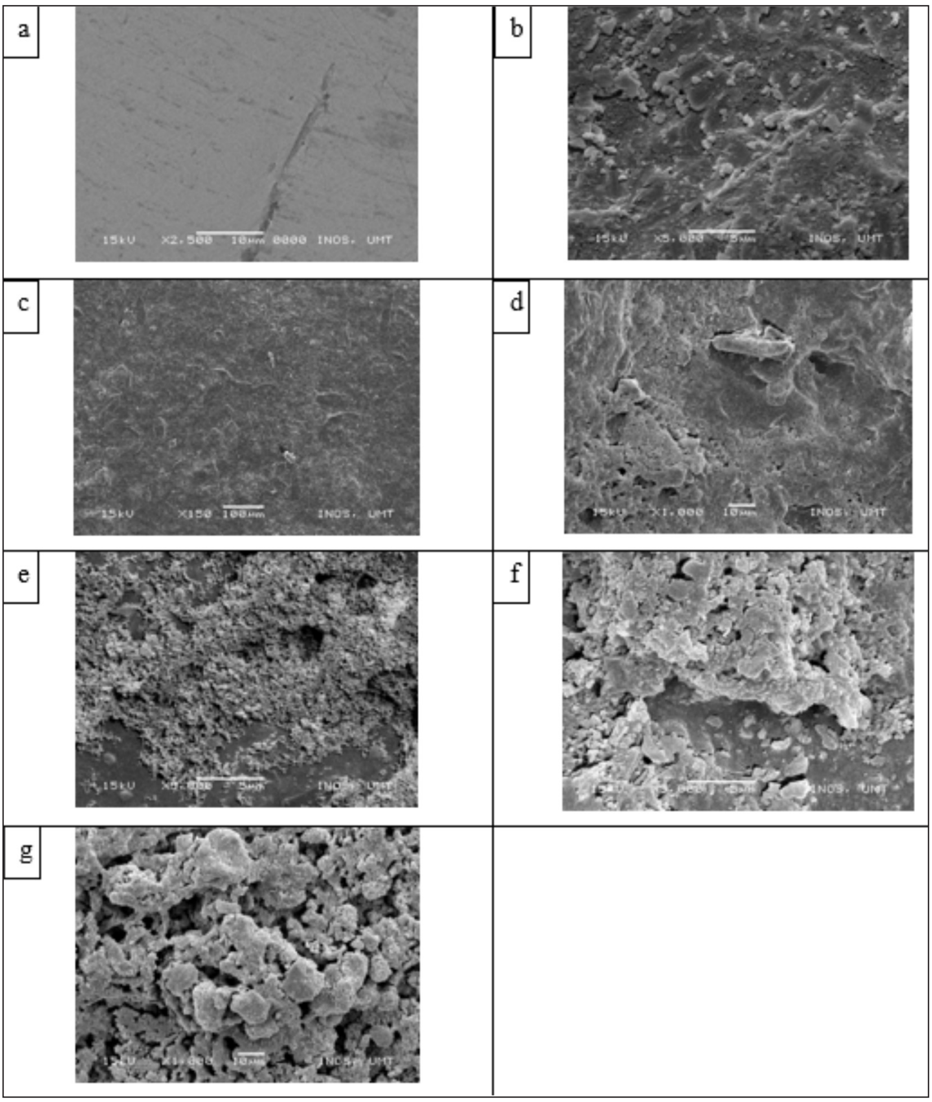

Figure 9: Surface analyses by SEM (a) uncoated mild steel surface, (b) without inhibitor after 10 days, (c) with inhibitor after 10 days, (d) after 20 days, (e) after 30 days, (f) after 40 days, (g) after 50 days

immersion in seawater. Formation of corrosion on the surface is clearly seen. In contrast, after 10 days immersion, the metal coupons coated with lemongrass extract, as shown in Figure 9 (c), exhibited minimal corrosion products and pits on the steel surface by forming a passive layer on the surface. This layer acts as a barrier towards corrosive ions on the surface of mild steel and the corrosion rate eventually decreased (Yetri *et al*., 2015; Verma *et al*., 2018). In addition, Figures 9 (d), (e), (f) and (g) show the surface morphology of mild steel in the presence of inhibitor for days 20, 30, 40 and 50, respectively. The rusting product formation can be seen throughout the immersion test.

The presence of lemongrass extract makes the surface remain smooth and with uniform deposits. In Figure 9 (d), the surface was smooth and no cracks were observed. However, in Figure 9 (e), many cracks were found and the surface became very rough as the immersion period increased. By day 40 onwards, as shown in Figures 9 (f) and Figure 9 (g), the surface became decidedly rougher and damage was observed in every part. Serious damage on the surface of mild steel was observed on day 50, Figure 9 (g), where the corrosion products have covered the surface of mild steel and formed a non-conductive layer (Hajar *et al*., 2016). Increasing the immersion period caused the rusting to progressively increase as the inhibition efficiency of lemongrass was inversely proportional to the immersion period. It was observed that there were significant improvements on the surface of mild steel in the presence of the inhibitor. The coated mild steel without the additive was in a much corroded state compared to the mild steels with the additive. This could be due to the formation of a protective film by the adsorption of inhibitor on the mild steel, thus inhibiting the corrosion (Helen *et al*., 2014; Mouaden *et al*., 2018; Onyeachu *et al*., 2019).

Conclusion

Lemongrass was extracted and incorporated into the protective layer, where further studies were conducted. FTIR spectroscopy analysis showed the presence of hydroxyl (OH) and carbonyl (C=O) in lemongrass extract. The result from the UV-Vis spectrum for ethanol solvent also shows the maximum wavelength was at 273.50 nm. The anti-corrosion properties of lemongrass have also been determined by conducting EIS and measuring weight loss. In this study, 20% lemongrass extract was found to be the best concentration since it has the highest value of charge transfer resistance (R_{ct}) which was 3.2224 x 10^6 Ω.cm^2, with the lowest double layer capacitance (C_{dl}) value of 0.2712 μF/cm^2. It was also found that untreated mild steel coupon has the highest percentage of weight loss compared with mild steel coated with 5%, 10%, 15% and 20% lemongrass extract during the immersion period. This research has also been able to determine the morphology of coated mild steel with the absence and presence of the additive by using optical and scanning electron microscopy. It was observed that there were significant improvements on the surface of mild steel in the presence of the additive compared to without the additive that was in a much corroded state during the immersion test.

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