



# Green-based corrosion protection for mild steel in 3.5% NaCl and distilled water medias: *Jatropha curcas* and Roselle extracts

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## Abstract

In this study, the natural sources of *Jatropha curcas* and *Hibiscus Sabdariffa calynx* (Roselle) extracts as green corrosion inhibitors were employed to improve the corrosion resistance of mild steel in simulated (3.5% NaCl) and distilled water medias. The inhibiting performance of two types of extracts was studied by visual inspection, microscope analysis, and various electrochemical measurements and the data of corrosion rate were statistically analyzed. The main phenolic compounds of *Jatropha* and Roselle extracts were confirmed via a liquid chromatography-mass spectrometry. Meanwhile, the functional groups of active phytochemical compounds were characterized via attenuated total reflection spectroscopy (ATR). The results of different electrochemical tests were revealed that the additions of *Jatropha* and Roselle extracts into the corrosion solution have an efficient corrosion inhibitor in reducing the corrosion rate though forming an adsorptive protective film on the surface, which reduced contact between samples' surfaces and solution medium and ultimately reduced corrosion attack. Practically, *Jatropha* extract showed the highest inhibitive efficiencies compared to Roselle extract in both simulated water and 3.5% NaCl solutions.

## 1. Introduction

Metal corrosion is an aggressive method that produces significant expenses by changing the metallic structure and the characteristics of the local setting. This is triggered by collateral responses such as the formation of oxides, the diffusion of metal cations into the metals matrix, and changes in local pH and electrochemical potential [1]. Carbon steel is the most common material used in constructing components and structures in the oil and gas industries, however, carbon steel corrosion has become a major issue in sectors that has resulted in a large amount of economic loss [2]. Corrosion is a constant and continuous problem thus it is difficult to achieve complete elimination of the problems. Rather, it would be more practical and achievable to apply corrosion preventive measures [3]. In constructing transmission pipelines, corrosion problems must be taken into considerations as it may have a detrimental impact on economic and safety aspects. One prevention method commonly used in industries especially in oil and gas industries is the application of corrosion inhibitors. Corrosion inhibitor is defined as a chemical substance that will cause a reduction of corrosion rate to an acceptable level upon addition to an environment [4]. Commonly, inhibitors are added in small amount counted as parts per million to the corrosive environment of a closed system, in which it is considered as a practical, economical and simpler alternative compared to replacing all of the structures in the respective

environment with high-cost corrosion resistance materials [5,6].

Up to date, there are many researches done to explore the possibility of using natural sources as green corrosion inhibitors mainly using plants and fruits extracts such as roselle extracts, and henna extracts. Benali *et al.* [7] have reported corrosion inhibitor using extraction of *Chamaerops humilis* plant with the presence of tannin compound that achieved inhibition efficiency of 86.51% when mild steel was immersed in 0.5M sulphuric acid. In other work done by Ibrahim and Habbab [8], extraction of eggplant peels as corrosion inhibitor for mild steel in 2M hydrochloric acid showed excellent inhibition properties. Agrawal [9] also proved that lemon peel attained 85.19% inhibition efficiency when 5%v/v of the extract was added for mild steel in 1M of HCl. Similarly, Akalezi *et al.* [15] studied the use of aqueous coffee powder extraction to inhibit the corrosion of mild steel in 1M HCl in which the results showed excellence inhibitive performances. In other words, plant-based inhibitors have great potential to be used to mitigate corrosion as well as having the advantage of being environment-friendly. One of the emerging potential sources of natural corrosion inhibitors is an extract of *Jatropha curcas* plants [10]. The latest development of research on *Jatropha curcas* has seen its potential to be developed into biodiesel which has advantages in terms of its renewability, produce fewer polluting substances than conventional diesel as well as the availability of the sources [11,12].

Following the success of producing biodiesel from *Jatropha curcas* sources, several researches have been made to explore the possibility of using *Jatropha curcas* as natural corrosion inhibitors. Nevertheless, specific researches focusing on the performance comparison between *Jatropha curcas* inhibitors and inhibitors made from different natural sources are still scarce. Therefore, this study aims to compare the inhibition efficiency of inhibitor made from *Jatropha curcas* extract with another plant-based inhibitor namely, *Hibiscus Sabdariffa calyxn* (Roselle) extract on corrosion behavior of mild steel in simulated sea- and distilled water medias.

## 2. Experimental procedure

### 2.1. Materials

In this study, the *Jatropha Curcas* leaves and Roselle (*Hibiscus Sabdariffa*) flower buds were collected from Herbal Valley, located at Guar Perahu, Pulau Pinang, Malaysia. The natural plants were washed separately using water and dried for 5 days, then followed by ground them into a fine powder using a mechanical grinder. Then, the weighed amount (700 g) was inserted into a round flask of a rotary evaporator and 5 liters of ethanol was added. The mixture was subjected to the rotary evaporator for 3 h at 70°C. After the process was completed, the mixture was subjected to the rotary evaporator for further one h in order to further concentrate the solution. Ethanolic extract of the plants was then obtained by filtering the concentrated solutions. The extraction process aimed to extract active chemical compounds from the plants as corrosion inhibitors which were performed based on the method reported by Sunday *et al.* [10]. A 25 mm diameter of mild steel was used as the working electrode, having the chemical composition (wt%) of C (0.13), Mn (0.48), P (0.02), Mo (0.01), Cr (0.11), Co (0.01) and Fe balance.

### 2.2. Chemical characterization of *Jatropha* and Roselle extracts

#### 2.2.1 2,2-diphenyl-1-picrylhydrazyl (DPPH) antioxidant test

The DPPH test was conducted using the same technique reported by Scherer and Godoy [19]: fifty microliters of different *Jatropha* and Roselle extract levels were added to 5 ml of a 0.004 percent DPPH (Sigma) methanol solution. The absorbance was read at 517 nm against a blank after an incubation period of 30 min at room temperature. Inhibition% (I%) of DPPH has been calculated as follows:

$$\text{Scavenging Activity (\%)} = \frac{A_c - A_s}{A_c} \times 100 \quad (1)$$

Where,  $A_c$  and  $A_s$  are the absorbance of sample and control, respectively. The metal inhibition is concentration-dependent and the concentration of the extract providing a 50% inhibition ( $IC_{50}$ ), which it is calculated from the graph plotted inhibition ratio against the concentration of the extract. The test was performed in a triplicate.

#### 2.2.1 Attenuated Total Reflection (ATR)

The *Jatropha* and Roselle extracts were characterized by using Attenuated Total Reflection (ATR) Spectroscopy in wavenumber ranging from 650  $cm^{-1}$  to 4000  $cm^{-1}$  IR spectrums obtained were used to identify the functional groups of active phytochemical compounds that present in the extract.

#### 2.2.2 Gas Chromatography-Mass Spectrometer (GC-MS)

GC-MS were performed using Agilent Technologies 7820A GC system and 5977E MSD provided with helium gas as carrier gas with a flow rate of 1  $ml \cdot min^{-1}$ . Injector temperature was set at 250°C while the interface temperature was set at 260°C with 4 min solvent delay. Temperature column increased gradually within the rate of 5°C·min<sup>-1</sup> and 10°C·min<sup>-1</sup> for the range of 40-240°C and 240-270°C, respectively. The initial oven temperature was set at 50°C and held for 3 min then increased 250°C at a rate of 5°C·min<sup>-1</sup> at held for 15 min. The scanning range was set at 50-500  $m \cdot z^{-1}$  with ionization energy of 70 eV [13].

### 2.3 Corrosion tests

#### 2.3.1 Preparation of corrosion test media

The corrosion test media for both immersion and electrochemical tests were distilled water and simulated seawater (3.5% NaCl) with and without the addition of plants extracts as inhibitors. Simulated seawater was prepared by adding 3.5% of sodium chloride into distilled water at room temperature. The amount of inhibitors were added based on the results obtained from the DPPH test. Table 1 shows the amount of extracts added for immersion and electrochemical tests. Immersion test was performed with the addition of both plants extracts in the amount stated without any subsequent addition throughout the immersion duration. For an electrochemical test, extracts addition was performed at the start of the experiment and the mild steel samples were immersed for 2 h to achieve the stable potential.

**Table 1.** Amount of extracts added for immersion and electrochemical tests.

	Inhibitor	Inhibitor concentration (ppm)	Mass of inhibitor (g·l)	Type of test solution	Total test solution
<b>Immersion test</b>	No Inhibitor	None	None	Distilled water 3.5% NaCl (Simulated Seawater)	5 L of Distilled water 5 L of 3.5% NaCl solution
	Jatropha Extract	4000	4	Distilled water 3.5% NaCl (Simulated Seawater)	20.00 g of extract + 5 L of Distilled water 20.00 g of extract + 5 L of 3.5% NaCl solution
	Roselle Extract	4000	4	Distilled water 3.5% NaCl (Simulated Seawater)	7.40 g of extract + 5 L of Distilled water 7.40 g of extract + 5 L of 3.5% NaCl solution
	No Inhibitor	None	None	Distilled water 3.5% NaCl (Simulated Seawater)	200 mL of Distilled water 200 mL of 3.5% NaCl solution
	Jatropha Extract	4000	4	Distilled water 3.5% NaCl (Simulated Seawater)	0.8 g of extract + 200 mL of Distilled water 0.8 g of extract + 200 mL of 3.5% NaCl solution
	Roselle Extract	4000	4	Distilled water 3.5% NaCl (Simulated Seawater)	0.296 g of extract + 200 mL of Distilled water 0.296 g of extract + 200 mL of 3.5% NaCl solution

**2.3.2 Electrochemical test and EIS analysis**

The electrochemical test or known as potential polarisation method has been conducted in a standard three-electrode cylindrical glass. Three electrodes include a working electrode (steel sample), counter electrode (carbon electrode) and reference electrode (saturated calomel electrode) in 250 ml of electrolyte (immersed medium) to complete the circuit. Polarisation was performed in accordance with the standard ASTM G5-94 by using computer-controlled VersaSTAT 3 software. The Tafel polarisation scan performed was at the potential of ±250 mV, with a potential scan rate of 0.333 mV·s<sup>-1</sup>, to achieve a current steady-state. The working electrode, which was the steel sample, was immersed in the immersion medium at an open circuit potential for 120 min before performing the electrochemical test. The polarization resistance (R<sub>s</sub>) was determined at an overvoltage lower than ±20 mV. The electrochemical measurements were determined in the frequency range between 100 kHz to 10 mHz and with a peak-to-peak amplitude (AC signal at an open circuit) of 5 mV. The inhibitor efficiency (%IE) was calculated using the following equation [14,15]:

$$\% IE = \frac{R_{ct} - R_{ct0}}{R_{ct}} \times 100 \quad (2)$$

where R<sub>ct0</sub> represents the charge transfer resistance without the inhibitor and R<sub>ct</sub> represents the charge transfer resistance with the inhibitor. The result of EIS

is the impedance of the electrochemical system as a function of frequency which was analyzed using *Zsimpwin* software. The EIS was performed in a frequency range of 0.01 to 100000 Hz.

**2.4.2 Immersion test**

The immersion test is commonly used to determine the corrosion rate of the metal by weight loss. This method is relatively easy and low cost because it does not require any equipment during the immersion process. This immersion test was conducted in accordance with ASTM G31-72 (laboratory immersion corrosion testing of metal) for a maximum duration of eight weeks. The mild steel samples were prepared before the immersion test. They were cut and then ground and cleaned to remove any oxides and contaminants on the surface. The prepared samples were tied with a plastic string so they could be hung in the immersion tank. The areas at the edge of the samples were covered with silicone so they would not be exposed to the test solution, and the exposed area calculated would only be the top and bottom surfaces of the samples. The steel samples were immersed in 5-liter glass containers, where all four containers were represented as a medium solution of distilled water or 3.5% NaCl solution. *Jatropha* and *Roselle* extract which acted as an inhibitor was added into the containers at different concentrations of 3000, 8000

and 10000 ppm. The low-carbon steel samples were weighed before the immersion test. They were immersed in the corrosion media where each separate media represented the 1, 2, 4, 6 and 8 weeks as immersion durations. Each steel sample was removed from the immersion tank following the duration period. The samples were then cleaned according to ASTM-G1 and reweighed. The immersed area was observed using a scanning electron microscope (Hitachi S-3400N VP) with energy-dispersive X-ray spectroscopy (EDX). The corrosion rate after the immersion test was determined using the following equation [16]:

$$\text{Corrosion rate (mm/year)} = \frac{(K \times w)}{(A \times t \times D)} \quad (3)$$

where K is a constant ( $8.76 \times 10^4$ ), w is the weight loss in grams, A is the exposed area, t is the immersion time in h and D is the density in  $\text{g}\cdot\text{cm}^{-3}$ . Corrosion rates without and with an inhibitor can determine the % IE by using the following equation [16]:

$$\% \text{ IE} = \frac{W_0 - W_{\text{corr}}}{W_0} \times 100 \quad (4)$$

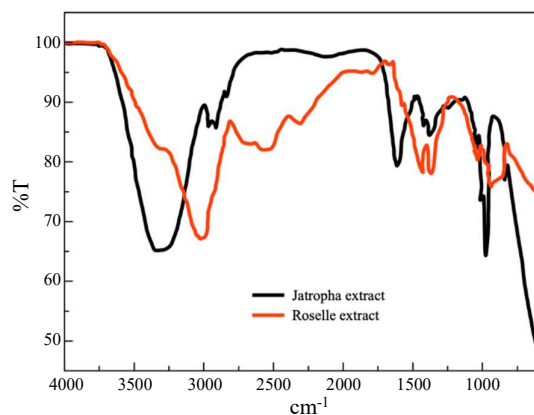
where  $W_0$  is the weight loss in the absence of the inhibitor and  $W_{\text{corr}}$  is the weight loss in the presence of the inhibitor in grams. The results were statistically analyzed using commercial statistical software namely, JMP 14.3.0 with implementing a graph builder mode. The level of significance was  $p < 0.05$ .

### 3. Results and discussion

#### 3.1 Characterization of the inhibitor passive layer

##### 3.1.1 ATR analysis

Figure 1 shows that both IR spectra illustrated broad peak at a wavenumber of  $3300\text{-}3400 \text{ cm}^{-1}$  which indicated the presence of OH functional groups chemical compound such as flavonoids and carboxylic acids, as reported by Molina *et al.* [17]. Strong peaks at wavenumber ranging from  $2800$  to  $3000 \text{ cm}^{-1}$  indicated the presence of C=O which is part of the flavonoid chain [18]. The peak at a wavenumber of  $1731 \text{ cm}^{-1}$  observed in IR spectrum of Roselle extract indicated C=O stretch [19]. On the other hand, peak indicating similar C=O stretch was observed at a wavenumber of  $1647 \text{ cm}^{-1}$  in Jatropha extract [20]. Therefore, the presence of various functional groups shown in Figure 1 proved that both Roselle and Jatropha extract contained chemical compounds such as alkaloids, flavonoids and organic acids that would provide a site of adsorptions for inhibition mechanism.



**Figure 1.** IR Spectrum of Jatropha and Roselle extracts.

#### 3.2 Compositional analysis of plant extracts using GC-MS

Table 2 shows the percentage of composition chemical compounds that were present in the plant extracts, in which it shows that all of the compounds have polar functional groups and hydrophobic hydrocarbon chains which are vital for inhibition mechanism of the respective plant extracts. Polar chemical compounds would serve as the site of adsorption on the metal surface and be capable of forming a continuous layer on the metal surface due to presence of polar functional groups.

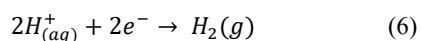
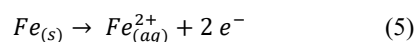
**Table 2.** Chemical compounds of Roselle and Jatropha extracts.

Extract	Name of Chemical Compound	Percentage (%)
Roselle	3-Amino-2-cyclohexenone	39.20
	2-Chloropent-4-ene carboxylic acid	24.71
	Dodecanoic acid	0.15
	aR-Turmerone	0.90
	9-Octadecanoic acid	0.99
	Isosteviol	6.03
	3,4-Dihydro-2H-pyran-2-carboxylic acid	5.43
	Succinic acid	17.86
	Cyclodecasiloxane	4.73
Jatropha	Copaene	0.44
	Caryophyllene	0.41
	Glucopyranose	1.33
	Neophytadiene	0.44
	Benzenepropanoic acid	0.49
	Benzenamine	16.88
	Isosteviol	75.53
	Squalene	4.48

### 3.3 Electrochemical behavior

#### 3.3.1 Potentiodynamic polarization

The PDP curves of the mild steel incorporated with different inhibitors and performed in different mediums are typically shown in Figure 2a and b. From Figure 2 (a), it can be seen that the shape and position of the PDP curves in the distilled water were significantly different from the presence of the inhibitors. Based on the Tafel fitting, it was found that the corrosion potential  $E_{\text{corr}}$  were slightly changed while the corrosion current densities  $i_{\text{corr}}$  was remarkably decreased from 5.76 to 1.317  $\mu\text{A}\cdot\text{cm}^{-2}$ . This observation showed the steel sample was possibly provided a comprehensive passive film that leads to affect the anodic and cathodic parts of PDP curves. Thus, the results that the reaction between the anode and cathode was inhibited by the adsorption of the inhibitor on the surface of mild steel samples. The main anodic dissolution of iron during the corrosion process along with a cathodic evaluation of  $\text{H}_2$  is represented by the following reactions, respectively:



On the other hands, the results of the Tafel fitting indicated that the existence of the *Jatropha* as inhibitor has more pronounced decreases in the current density associated with the formation a better protection layer, thus reduce the corrosion rate remarkably [21,22]. This is kind of enhancement may also attribute to the adsorption of the inhibitor molecules on the surface of mild steel [23], has been also reported by Amita and Bharathi [24] that the addition of the green inhibitor may contain a phytochemical constituent. This is would be acted as physical barriers that restricted the diffusion of ions and therefore, prevent the surface metal from a massive corrosion attack. Furthermore, the addition of both *Roselle* extract and *Jatropha* extract independently shifted the anodic and cathodic curves to more positive region [25], as shown in Figure 2 (a). Therefore, it can be alleged that both *Roselle* extract and *Jatropha* extract were a mixed-type inhibitor. Mixed-type inhibitors inhibit corrosion attack by forming an adsorptive protective layer on the surface of metal without any preference to either anodic or cathodic site [26]. The results showed that addition of *Roselle* extract and *Jatropha* extract reduced the corrosion rate from 0.06702 mmpy to 0.03096 mmpy and 0.01532 mmpy, respectively. Comparing between the two plant extracts, it can be seen that the addition of *Jatropha* extracts reduced corrosion rate more significantly than *Roselle* extract in the distilled water environment. With referring to Table 3, it can be revealed that the *Jatropha* extract addition achieved inhibitor efficiency of 79.83% compared to *Roselle* extract addition which achieved

inhibition efficiency of only 53.80%. Overall, the addition of *Roselle* and *Jatropha* extract was capable of reducing corrosion attack in distilled water environment with *Jatropha* extract being able to inhibit corrosion more significantly compared to *Roselle* extract. Also, it should be noted that the results obtained were in agreement with the results attained from the immersion test in the subsection 3.3. To provide an extensive investigation on the behavior of proposed inhibitors, the PDP curves of the mild steel with and without the existence of inhibitors in the 3.5% NaCl solution are exhibited in Figure 2 (b). It can be seen the  $i_{\text{corr}}$  of the mild steel had a higher value in the acidic solution of 19.041  $\mu\text{A}\cdot\text{cm}^{-2}$  and been reduced to 3.557  $\mu\text{A}\cdot\text{cm}^{-2}$  with the addition of *Roselle* extract, whereas, the addition of *Jatropha* extract had reduced it 1.855  $\mu\text{A}\cdot\text{cm}^{-2}$ . The reduction of the current density was caused by the formation of protective film on samples' surfaces by adsorption of active phytochemical compounds present in the respective plant extracts. Shifting of both anodic and cathodic curves by addition of *Roselle* extract and *Jatropha* extract indicated that these plant extracts were mixed-type inhibitors when added in 3.5% NaCl solution. As stated by Ameer & Fekry [27], the mixed-type inhibitors acted by blocking anodic and cathodic sites on samples' surfaces which result in a reduction of surface area for corrosion attack. Based on the tabulated data in Table 3, it shows that corrosion rate was also being reduced by the addition of *Roselle* extract and *Jatropha* extract in which corrosion rate of 0.22145 mmpy obtained from sample void of inhibitors was reduced to 0.04137 mmpy and 0.02158 mmpy, respectively. The reduction of corrosion rate occurred due to formation of protective film on the samples surfaces by adsorption of active chemical compounds such as tannin, saponin, alkaloid, flavonoid and carboxylic acids which were a presence in these plant extracts [28]. Comparing between the two plant extracts, it can be seen that addition of *Jatropha* extract reduced corrosion rate more significantly compared to *Roselle* extract. This declared that the *Jatropha* extract recorded inhibition efficiency of 90.26% while *Roselle* extract recorded inhibition efficiency of 81.32% when added to 3.5% NaCl solution. Overall, both addition of *Roselle* extract and *Jatropha* extract attained high inhibition efficiencies in 3.5% NaCl solution. However, both plant extracts showed inhibition capabilities in distilled water as well as a 3.5% NaCl solution. It should be noted that the corrosion rate of mild steel samples with the addition of the extracts was higher in 3.5% NaCl solution compared to the distilled water environment. Chloride ions presence in 3.5% NaCl solution facilitated corrosion attack on the samples surfaces thus causing less material loss compared to distilled water. This phenomenon was due to the fact that chloride ions were a presence in NaCl solution. The chloride ions assisted the adsorption process of active chemical compounds present in the plant

extracts on the samples' surfaces by creating bridges between the sample surface and active chemical compounds [29]. Similar results were reported by Ridhwan *et al.* [30] in which the addition of KCl improved performances of plant-based inhibitors. Thus, more adsorption processes occurred and resulting in higher surface coverage of protective films formed. As more surface was covered by protective films, corrosion attack was reduced as these films created a barrier to prevent contact between aggressive solution medium and the samples' surfaces. Therefore, the plant extracts were capable of reducing corrosion rate at a greater extent and achieving higher inhibition efficiencies in 3.5% NaCl solution compared to in distilled water. Lastly, the results of electrochemical test in an agreement in line with immersion test in which both extracts showed higher inhibitive performances in 3.5% NaCl solution. In addition, the Jatropha extract showed the highest inhibitive efficiencies compared to Roselle extract in both distilled water and 3.5% NaCl solution.

### 3.3.2 EIS analysis

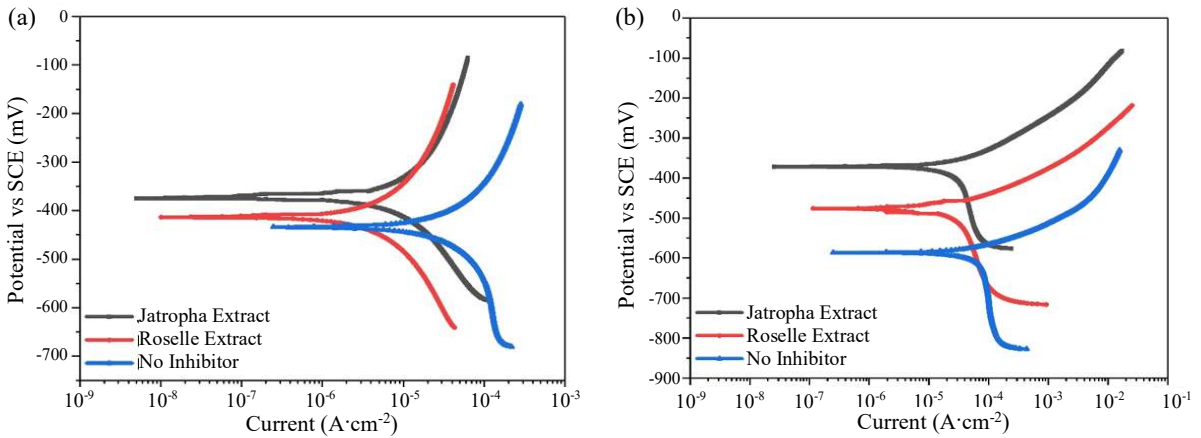
EIS analysis was performed using mild steel samples as a working electrode in distilled water with and without of Roselle and Jatropha extracts addition as corrosion inhibitors. Figures 3 (a) and 3 (b) show the Bode and Nyquist plots and the extracted data were tabulated in Table 4. The Bode plot in Figure 3a shows that there was an increment of electrical impedance when Roselle and Jatropha extracts were added into distilled water environment which can be seen from the increment of highest magnitude value of bode plots shown. Addition of Jatropha extracts recorded the highest impedance magnitude at the value of approximately  $290 \Omega \cdot \text{cm}^2$ . While, the addition of Roselle extract and sample without inhibitor recorded impedance value of approximately 270 and  $120 \Omega \cdot \text{cm}^2$  respectively. Increment of electrical impedance was due to the presence of the adsorptive film on the surface of the immersed samples in which the film resisted charge transfer. Also, Nyquist plots with and without the addition of inhibitors were illustrated as semi-circle shape which indicated resistive behavior [27], as shown in Figure 3 (b). Addition of Roselle extract and Jatropha extract did not change the shape of the Nyquist plot but rather only increased the diameter of the semi-circle plots. This indicates that the addition of both Roselle and Jatropha extracts did not change the electrochemical reactions responsible for corrosion and inhibition occurred only through its adsorption on the surface of the metals samples [31]. According to the EIS fitting parameters measurements in Table 4, it was revealed that there is an increment of charge transfer resistance ( $R_{ct}$ ) and decrement of layer capacitance ( $C_{dl}$ ) when respective plant extracts were added into solution medium as corrosion inhibitors. Addition of Roselle extract exhibited increment of  $R_{ct}$  value from  $72.25$  to  $113.10 \Omega \cdot \text{cm}^2$ . Correspondingly, the addition of Jatropha

extracts increased  $R_{ct}$  value from  $72.25 \Omega \cdot \text{cm}^2$  (obtained from control setup) to value of  $159.40 \Omega \cdot \text{cm}^2$ . Increment of  $R_{ct}$  value was due to the presence of the adsorbed film on the samples' surface which increased the resistance to charge transfer. Higher  $R_{ct}$  value indicated higher surface coverage of protective film that has been formed on the samples' surfaces. Also, addition of Roselle extract and Jatropha extract reduced  $C_{dl}$  value from  $2.325 \times 10^{-3} \text{ F} \cdot \text{cm}^2$  (obtained from control setup) to  $9.441 \times 10^{-4} \text{ F} \cdot \text{cm}^2$  and  $1.205 \times 10^{-4} \text{ F} \cdot \text{cm}^2$  respectively. As stated by Nazeer *et al.* [31], the reduction in  $C_{dl}$  value was due to higher attainment of surface coverage of protective film formed on the surface. This lead to reduce the interfacial capacitance as the inhibitor molecules with smaller dielectric constants replaced water molecules present on the samples' surfaces by adsorption. It can also be aforesaid that addition of Roselle extract and Jatropha extract inhibit corrosion through the formation of adsorptive protective film on the surface which reduced contact between samples' surfaces and solution medium and ultimately reduced corrosion attack. Presence of protective film was justified by an increment of  $R_{ct}$  value and decrement of  $C_{dl}$  value as revealed in the obtained curves. Comparing between the addition of Roselle extract and Jatropha extract in a distilled water environment. It can also be seen that Jatropha extract performed better in distilled water environment which was justified by a higher increment of  $R_{ct}$  value and decrement of  $C_{dl}$  value compared to Roselle extract. Thus, it concluded that addition of Jatropha extract was capable of inhibiting corrosion to a greater extent compared to the addition of Roselle extract in a distilled water environment. Also, these results were in line with the results of immersion test and electrochemical test discussed in earlier sections.

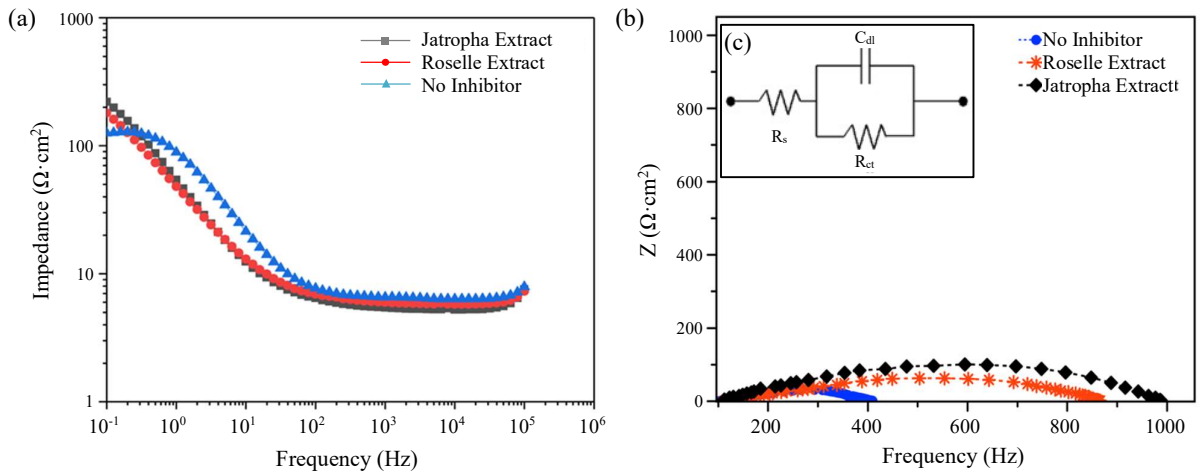
From Figure 4 (a) and 4 (b), it can be seen that the addition of both Roselle extract and Jatropha extract increased the electrical impedance value compared to the uninhibited sample. Jatropha extract attained the highest magnitude followed by Roselle extract and lastly uninhibited sample. Figure 4 (a) displays that the Jatropha extract addition attained highest magnitude value which was at approximately  $1050 \Omega \cdot \text{cm}^2$ . While, the Roselle extracts addition recorded highest magnitude value of approximately  $1000 \Omega \cdot \text{cm}^2$  and sample without inhibitor achieved the lowest value at  $900 \Omega \cdot \text{cm}^2$ . Increase in electrical impedance by addition of plant extracts indicated the presence of the adsorptive film on the samples surfaces in which the film increased resistance towards charge transfer. On the other hands, the Nyquist plots obtained in Figure 4 (b) showed semi-circle shape in which they indicated resistive behavior of the system [32]. The addition of both plant extracts did not change the shape of the plots, rather increased the diameter of semi-circle plots. Thus, the addition of the extracts as inhibitors did not alter electrochemical reactions of the corrosion rather inhibited corrosion by adsorption on the surface of samples. Based on the fitting parameter measurements

given in Table 5 shows that the addition of both Roselle extract and *Jatropha* extract increased  $R_{ct}$  and reduced  $C_{dl}$ . Moreover, it was exhibited also that the addition of Roselle extract and *Jatropha* extract in 3.5% NaCl solution medium increased  $R_{ct}$  value of  $37.30 \Omega \cdot \text{cm}^2$  (obtained from control setup) to  $254.30 \Omega \cdot \text{cm}^2$  and  $400.40 \Omega \cdot \text{cm}^2$ , respectively. The increasing  $R_{ct}$  value was due to the presence of protective films that were formed through adsorption processes, similar as reported in a study by [33]. Increment of  $R_{ct}$  value indicated higher surface coverage of the protective film formed which results in lower interaction between samples' surfaces and the solution medium. On the other hand,  $C_{dl}$  value decreased by addition of Roselle extract and *Jatropha* extract from the value of  $1.098 \times 10^{-3} \text{ F} \cdot \text{cm}^2$  to  $8.561 \times 10^{-4} \text{ F} \cdot \text{cm}^2$  and  $1.205 \times 10^{-4} \text{ F} \cdot \text{cm}^2$  respectively. Reduction of  $C_{dl}$  value was results of decreasing corrosion product formed on the sample's surface as the addition of Roselle extract and *Jatropha* extract reduced corrosion

attack. Similar observations were reported in a study by Ibrahim and Habbab [33]. The results obtained proved that the addition of Roselle extract and *Jatropha* extract have the capabilities of reducing corrosion attack through the formation of the adsorptive protective film. The presence of the protective film on samples surfaces was justified by increasing  $R_{ct}$  value and decreasing  $C_{dl}$  value. According to Zouarhi *et al.* [28], these occurrences were caused by adsorption of inhibitors' chemical compounds on samples' surfaces. Nevertheless, the addition of *Jatropha* extract result in a higher value of  $R_{ct}$  and lower the value of  $C_{dl}$  compared to the addition of Roselle extract in 3.5% NaCl solution which indicates that *Jatropha* extract showed higher inhibition capability in 3.5% NaCl solution compared to Roselle extract. These results were in agreement with results obtained from immersion test as well as the electrochemical test.



**Figure 2.** Tafel plots of mild steel in the absence and in the presence of inhibitors in (a) distilled water, (b) 3.5% NaCl solution.



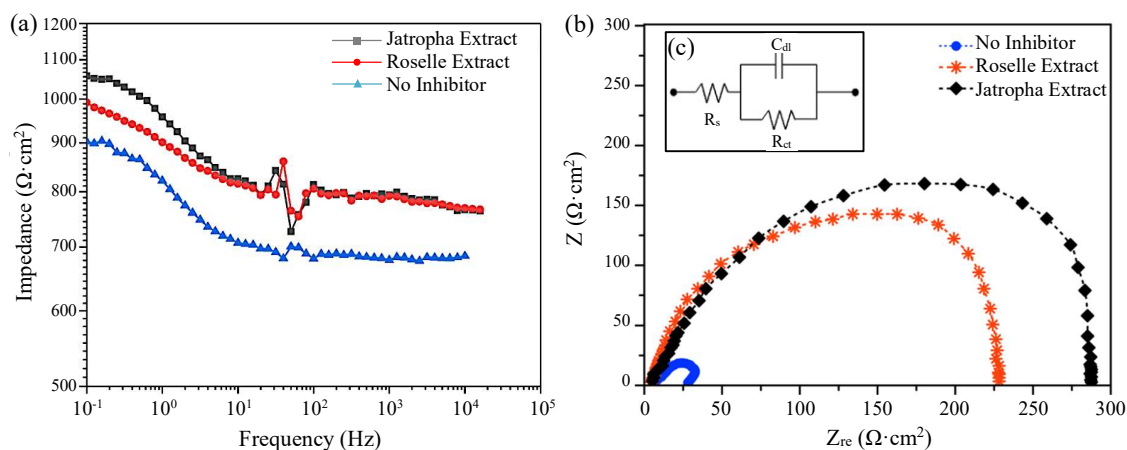
**Figure 3.** (a) Bode and (b) Nyquist Plot recorded for mild steel with and without the addition of inhibitors in distilled water (c) Randle's Circuit for impedance data fitting,  $R_s$  in series with a combination of layer capacitance,  $C_{dl}$  and charge transfer resistance,  $R_{ct}$  in parallel.

**Table 3.** Results of the electrochemical test performed in distilled water and 3.5% NaCl solution.

Medium	Types of Inhibitor	$E_{corr}$ (mV)	$i_{corr}$ ( $\mu\text{A}\cdot\text{cm}^2$ )	$\beta_a$ (mV·dec)	$\beta_c$ (mV·dec)	Corrosion Rate (mppy)	IE (%)
Distilled Water	No Inhibitor	-434.30	5.76	1247	1803	0.067	----
	Roselle Extract	-413.77	2.66	563.46	571.63	0.030	53.80
	Jatropha Extract	-372.82	1.31	100.17	131.82	0.015	79.83
3.5% NaCl solution	No Inhibitor	-622.51	19.04	100.78	10.01	0.221	----
	Roselle Extract	-469.91	3.55	46.89	63.55	0.041	81.32
	Jatropha Extract	-361.81	1.85	54.25	58.84	0.021	90.26

**Table 4.** EIS fitting parameters of mild steel with and without inhibitors in distilled water and 3.5% NaCl solutions.

Medium	Types of Inhibitor	The resistance of Solution, $R_s$ ( $\Omega\cdot\text{cm}^2$ )	Charge Transfer Resistance, $R_{ct}$ ( $\Omega\cdot\text{cm}^2$ )	Capacitance, $C_{dl}$ ( $\text{F}\cdot\text{cm}^2$ )
Distilled water	No Inhibitor	6.269	72.25	$2.325\times 10^{-3}$
	Roselle Extract	6.938	113.10	$9.441\times 10^{-4}$
	Jatropha Extract	6.887	159.40	$1.205\times 10^{-4}$
3.5% NaCl solution	No Inhibitor	5.710	37.30	$1.098\times 10^{-3}$
	Roselle Extract	6.269	254.30	$8.561\times 10^{-4}$
	Jatropha Extract	5.980	400.40	$1.205\times 10^{-4}$

**Figure 4.** (a) Bode and (b) Nyquist plot recorded for mild steel with and without addition of Inhibitors in 3.5% NaCl Solution, (c) Randle's Circuit for impedance data fitting,  $R_s$  in series with combination of layer capacitance,  $C_{dl}$  and charge transfer resistance,  $R_{ct}$  in parallel.

### 3.4 Immersion test

Corrosion rate signified the speed at which metals deteriorate in the environment. Therefore, a lower value of corrosion rate was preferable. The corrosion rate against immersion period is statistical analysis and plotted in Figure 5 accompanied by indicating the

Root Mean Square Error (RMSE) and relative measure of fit ( $R^2$ ). It was found that the corrosion rate of the samples without inhibitor in the distilled water was recorded at 0.105 mppy after 1 week of immersion and decreased with fluctuation to corrosion rate of 0.0812 mppy after 8 weeks of immersion. While for the samples immersed in 3.5% NaCl



solution medium, it can be seen that the corrosion rate decreased from 0.8661 mmpy which was recorded 0.2402 mmpy and 0.0544 mmpy for week 1 and 8 of immersion, respectively. The fluctuations may be caused by physical disturbances occurred and gravity which results in corrosion product to fall off from the surface as they did not adhere to the samples surfaces. Therefore, the amount of corrosion product presence on the samples surfaces reduced which also result in a reduction of barrier effect. Generally, the corrosion rate of samples without inhibitors showed a decreasing trend throughout the immersion period. This is due to the formation of corrosion product (ferrous hydroxide) which formed on the samples' surface which served as a corrosion barrier between samples' surface and solution medium. With increasing the immersion time, denser and thicker corrosion products have been formed, thus providing more barrier on the surface though they were not protective [34]. The barrier only slowed down the migration of ions and electrons between the anode and cathode region thus decelerating the corrosion process through a cumulative weight loss of sample without inhibitors addition was still high compared to inhibited samples. On the other hand, samples that have been immersed with the addition of Roselle extract and Jatropa extract as corrosion inhibitors showed a lower corrosion rate than samples with no inhibitor. From the same point of view, the corrosion rate exhibited a decreasing trend for samples immersed with the addition of Roselle extract and samples immersed with the addition of Jatropa extract at a longer immersion period. Reduction of corrosion rate was due to the formation of the adsorptive protective layer on the samples surfaces which inhibit contact between samples surfaces and distilled water solution medium [34]. At longer immersion period, more surface coverage can be attained thus result in a reduction of corrosion rate. Comparing the statistical analysis of both additions of Roselle extract and Jatropa extract as a corrosion inhibitor in a distilled water environment, it can be seen that Jatropa extract reduced corrosion rate to a greater extent compared to Roselle extract. However, both incorporations attained the best fitting with RMSE is equal to zero and the highest  $R^2$  in distilled water compared to a 3.5% NaCl solution. Higher reduction of corrosion rate by addition of Jatropa extract was due to higher amount of active phytochemical compounds presence in Jatropa extract thus increasing surface coverage of protective film formed on the sample surface [32].

### **3.5 3D Surface inspection**

Figures 6 (a) to 6 (f) shows the surface 3D visualization for the samples that have been immersed in distilled water and 3.5% NaCl Solution environment with and without the addition of inhibitors for 8 weeks of immersion period. The samples that were immersed in

distilled water in the absence of inhibitor contained a significant amount of corrosion products as shown in Figure 6 (a), which were reddish-brown deposits that adhered on the samples' surfaces. With the addition of Roselle extract, the surface sample exhibited dark slimy film is observed in Figure 6 (b). The dark film was due to adsorption of active chemical compounds that were present in the Roselle extract. On the other hand, samples that have been immersed in distilled water with the addition of Jatropa extract exhibited light grey slimy film present on the surface (see in Figure 6 (c)), there was little corrosion product formed on the sample's surfaces compared to samples that have been immersed without the addition of inhibitor. Figure 6 (d) shows that there was reddish-brown deposits presence on the surfaces of samples that were immersed in 3.5% NaCl solution without addition of inhibitor. These deposits were corrosion product that was formed due to reaction of the samples' surfaces with oxygen. However, the amount of deposits observed on the samples' surfaces reduced as immersion period increased. This is due to weak adherence of these deposits on the surfaces which caused them to fall off from the surfaces. As the deposits formed on the sample's surfaces were porous and non-protective, continuous contact with the environment enabled the corrosion process to continue. Over longer immersion period, accumulation of corrosion product on the surfaces caused them to fall off from the surface due to the effect of gravity. On the other hand, samples that have been immersed with the addition of Roselle extract as corrosion inhibitor had a little amount of deposits observed on the surfaces, as shown in Figure 6 (e). The amount of deposits slightly increased with increasing immersion period though the amount of deposits formed were still little. Moreover, the surfaces of the samples were covered in dark grey slimy film in color. Upon cleaning, there was a noticeable change of appearance of samples surfaces from shiny grey (before immersion) to darker grey (after immersion and cleaning).

Samples that were immersed with the addition of Jatropa extract as inhibitor contained no significance corrosion product formed on the surface, as displayed in Figure 6 (f). This was due to the formation of a protective film on the samples' surface. Prior to cleaning, the surfaces of the samples were covered with slimy light grey film on their surfaces. It was found that samples immersed in an environment with the addition of Jatropa appeared cleaner and shinier after cleaning than samples that were immersed without the addition of inhibitor. These observations proved that there were less or no significance corrosion process occurred throughout the immersion period. At longer immersion interval, there was a slight increment of deposits present on the samples surfaces though their amount was very small due to the formation of a stable protective layer.

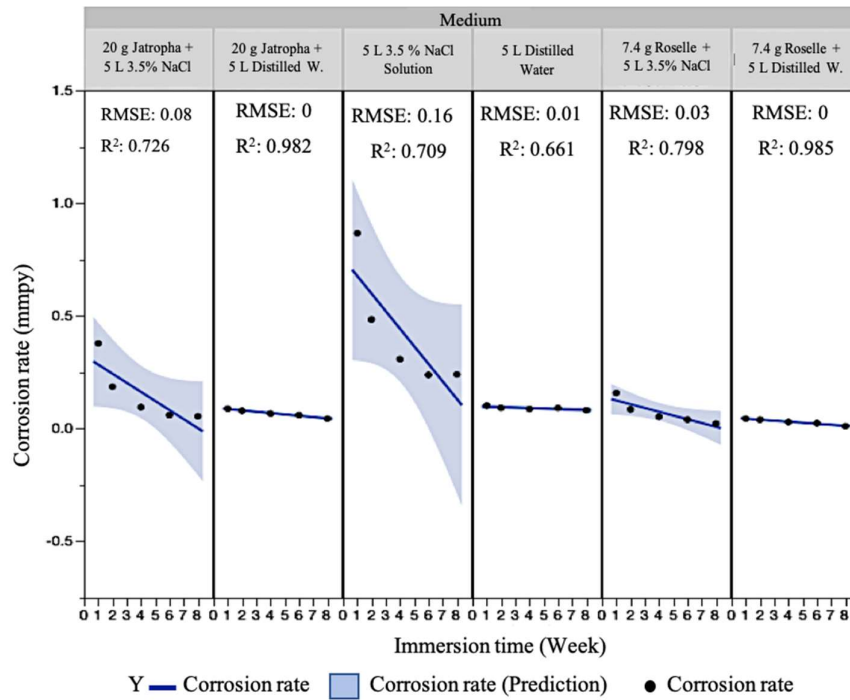


Figure 5. Corrosion rate vs. immersion time in distilled water and 3.5% NaCl solution with and without inhibitors addition.

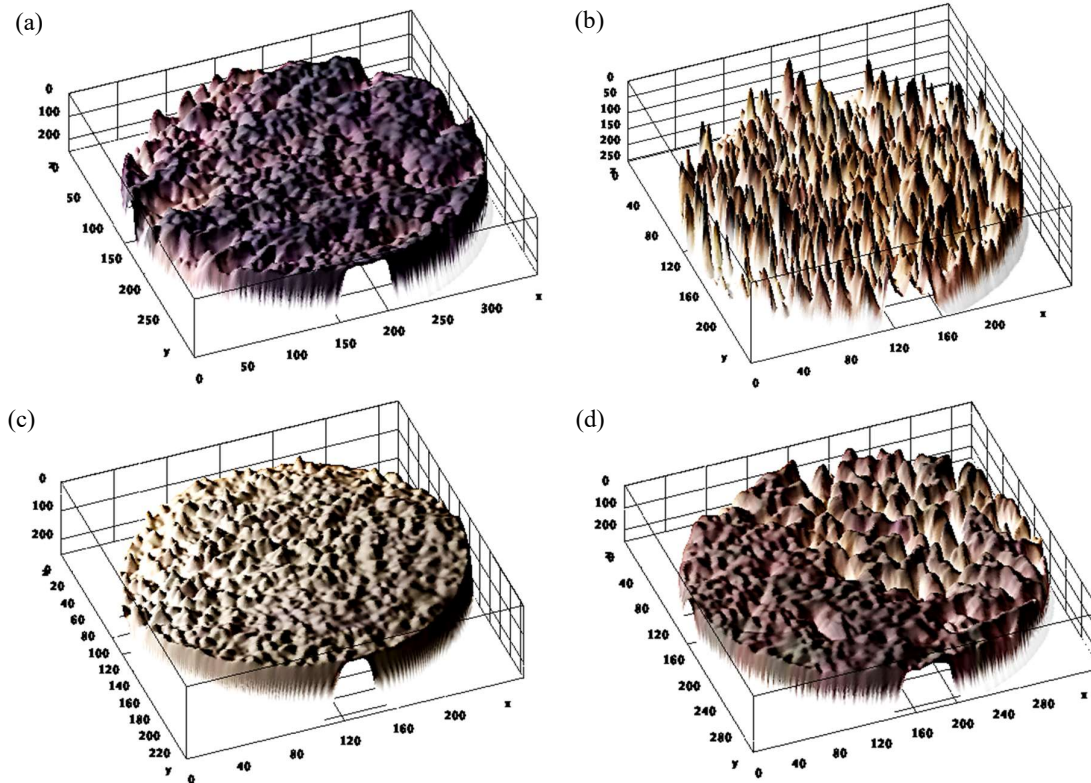
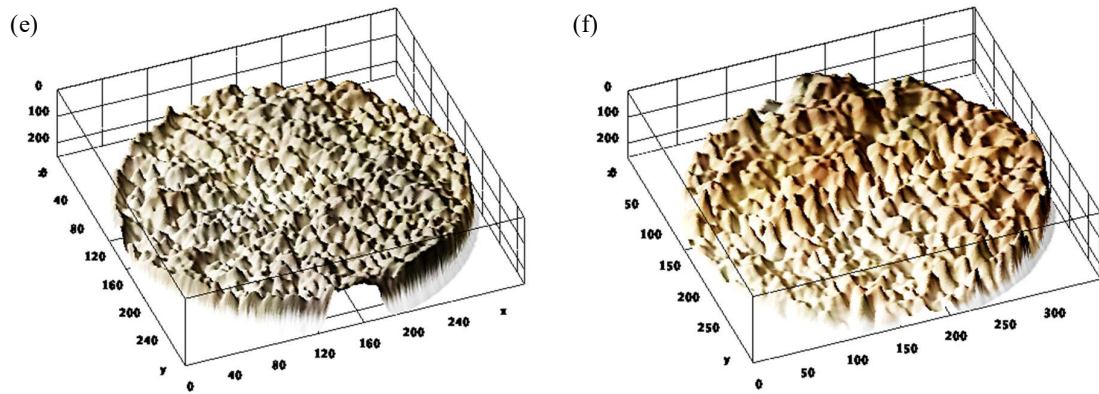


Figure 6. 3D visualization images of the immersed samples for 6 weeks with (a) No inhibitor/distilled water, (b) Roselle/distilled water, (c) Jatropa/distilled water, (d) No inhibitor/3.5% NaCl, (e) Roselle/3.5% NaCl, (f) Jatropa/3.5% NaCl.

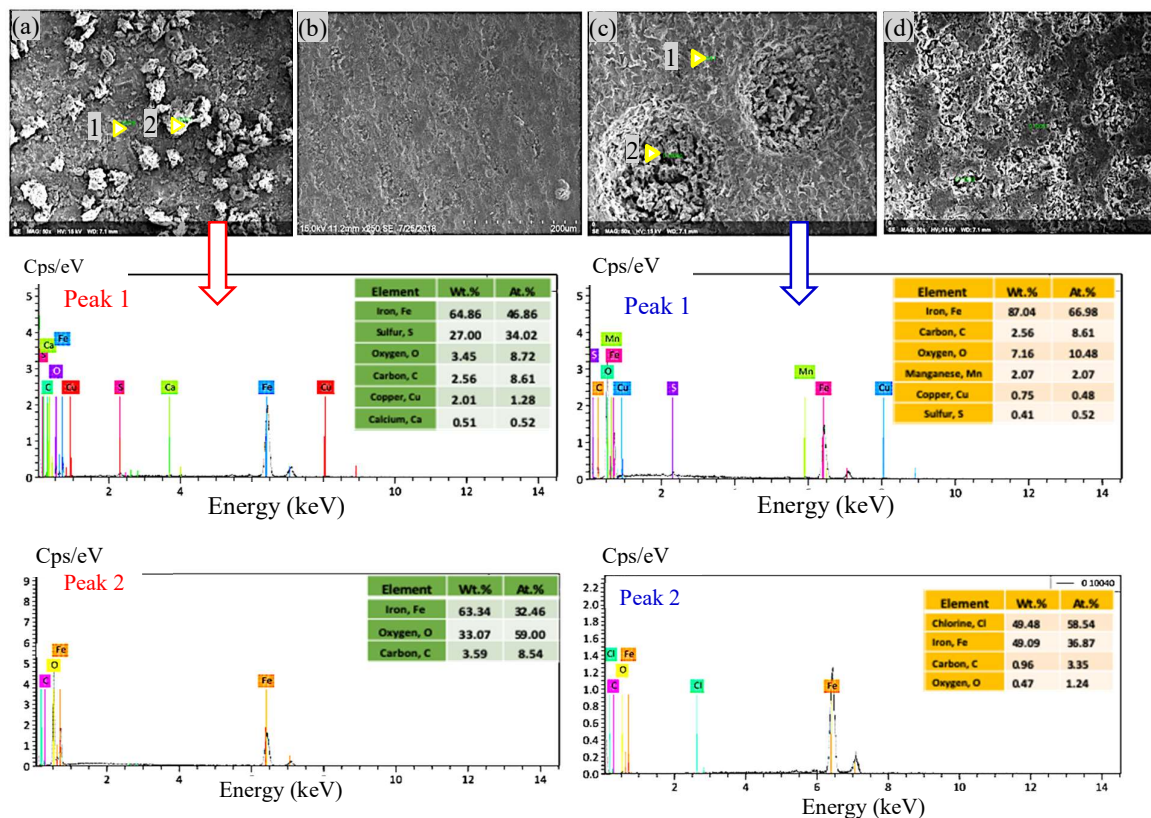


**Figure 6.** 3D visualization images of the immersed samples for 6 weeks with (a) No inhibitor/distilled water, (b) Roselle/distilled water, (c) *Jatropha*/distilled water, (d) No inhibitor/3.5% NaCl, (e) Roselle/3.5% NaCl, (f) *Jatropha*/3.5% NaCl. (continue)

### 3.6 Microscope analysis

Figure 7 (a) to 7 (d) shows an image of a sample immersed in distilled water and 3.5% NaCl without the addition of an inhibitor with corresponding EDS analysis performed on the surface. Figure 7 (a) shows that the surface of the sample appeared rough due to the uniform corrosive product that been formed on the surface of the immersed sample. EDS analysis shows that the high percentage of oxygen indicates that oxidation reaction has occurred between the sample surface and test solution medium thus forming corrosion product precipitates. Hussin and Kassim [35] stated in their study, an increment of oxygen content was due to formation of ferrous hydroxide (corrosion product) which was the result of the reaction of mild steel sample with oxygen present in distilled water. Besides that, EDS analysis performed on the surface of the sample showed that the sample had typical mild steel composition with an increase in oxygen content due to corrosion product formed when the metal corroded. The surface of the sample with the addition of Roselle extract is shown that the appearance of the surface was less rough compared to the surface of the sample immersed without the addition of inhibitor most probably due to less uniform corrosion occurred. EDS analysis performed on the sample surface exhibited a higher percentage of carbon (5.05 wt%) compared to 1.56 wt% carbon content of the sample before immersion. A higher percentage of carbon most probably due to the contribution of the hydrocarbon chain of active chemical compounds that adsorbed on the sample surface, forming a protective film [31]. The surface of the sample immersed with the addition of *Jatropha* extract showed less rough appearance compared to uninhibited sample which indicated that less uniform corrosion occurring on the surface, as shown in Figure 7 (b). The elemental analysis showed an even higher percentage of carbon of 9.60 wt%

compared to carbon content of 1.56 wt% in the sample before immersion. This is attributed to the adsorption of active chemical compounds on the surface as most of these chemical compounds contained hydrocarbon chains [35]. From Figure 7 (c), it can be seen that uniform corrosion has occurred on the sample. Small pit-like features that can be seen throughout the sample surface. Surrounding the pit-like features were rough surface caused by uniform corrosion occurred on the surface of the mild steel sample. A higher percentage of oxygen content observed was due to corrosion of sample surface with aggressive NaCl solution, forming corrosion product. EDS result shows the presence of iron, carbon, oxygen, and chlorine-based on the spectrum analyzed directly into the pit-like feature. Presence of oxygen and chlorine indicated that oxidation reaction has occurred in the area and the presence of chlorine facilitated the corrosion process. Image of the sample immersed with the addition of Roselle extract after 8 weeks of immersion time appeared rough due to uniform corrosion occurred. Nevertheless, the appearance of the surface was less severe compared to the surface of an uninhibited sample. Elemental composition confirmed that there was an increment of carbon content at 12.91 wt% compared to 1.56 wt% of carbon content before immersion was performed. This may related to presence of adsorbed active chemical compounds of Roselle extract which formed a protective film on the surface [19]. Similar observations were seen in sample immersed with *Jatropha* extract addition after 8 weeks immersion as shown in Figure 7 (d). The sample surface was rough as a result of uniform corrosion though the appearance was smoother than the uninhibited sample. The surface also showed higher carbon content than before immersion was performed due to contribution from adsorbed chemical compounds of *Jatropha* extract on the sample surface [36].



**Figure 7.** Immersion micrographs and their corresponding elemental composition analysis after 8 weeks Immersion of (a) No inhibitor/distilled water, (b) Jatropha/distilled water, (c) No inhibitor/3.5% NaCl, and (d) Jatropha/3.5% NaCl.

#### 4. Conclusions

Roselle and Jatropha extracts acted as an effective corrosion inhibitor for mild steel in both distilled water and 3.5% NaCl environment. The corrosion rate of both selected inhibitors decreased with increased immersion time. From the EIS plots, it is clear that the charge transfer resistance increased with the presence of Roselle and Jatropha extract, in addition, the inhibition efficiencies obtained from weight loss measurements were in good agreement with polarization and EIS methods. Jatropha extract exhibited higher inhibitive performances compared to Roselle extract, which was signified by lower corrosion rate and higher inhibition efficiency at 8 weeks of immersion in both distilled water and 3.5% NaCl environment. Inhibition efficiencies were calculated based on the results of the electrochemical test in which addition of Jatropha extract in 3.5% NaCl recorded the highest efficiency at 90.26% and the SEM studies showed the formation of inhibitor film on mild steel surface.

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