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ORIGINAL ARTICLE

The litchi (*Litchi Chinensis*) peels extract as a potential green inhibitor in prevention of corrosion of mild steel in 0.5 M H_2SO_4 solution

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B. EIS;
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C. Acid corrosion

Abstract The corrosion inhibition of mild steel in 0.5 M H_2SO_4 solution by the extract of litchi peel (*Litchi chinensis*) was studied by weight loss method, potentiodynamics polarization and electrochemical impedance spectroscopy (EIS). The results show that the litchi peels extract acts as mixed-type inhibitor. The inhibition of corrosion is found to be due to adsorption of the extract on metal surface, which is in conformity with Langmuir's adsorption isotherm. UV–Vis, Fourier transform infrared (FT-IR) spectroscopy and Scanning electron microscopy (SEM) studies confirm that the inhibition of corrosion of mild steel occurs through adsorption of the inhibitor molecules.

1. Introduction

Corrosion of raw materials in industries has been a perennial problem worldwide. In petrochemical industries, in particular, corrosion has been one of the major concerns since carbon steel is widely employed as raw materials for construction of pipelines in the oil and gas industries (Ghareba and Omanovic, 2010). The unexpected metal decomposition or eating away of metal has created the barrier to the growing industries. Therefore, the protection of metal or alloy from corrosion is of paramount importance. Many successful endeavours have been made to deliver methods for controlling the menace of corrosion. One of the methods is the corrosion inhibition by inhibitor in which corrosion inhibitor is used to retard the rate of corrosion of the metal or alloy. This method is considered to be one of the most practical, effective and viable methods (Oguzie et al., 2009; Binks et al., 2011; Tao et al., 2010; Abelev et al., 2007). The large number of organic compounds having hetero-atoms such as N, O, S atoms and π-electrons was reported as the efficient corrosion inhibitors under aggressive environments such as acidic or alkaline mediums etc. (de Souza and Sinelli, 2009; Fekry and Mohamed, 2010; Lyon, 2004; Fu et al., 2010; Sheng et al., 2007). They provide excellent inhibition and retard the rate of corrosion of metal by adsorbing itself on the surface of the metal (Migahed et al., 2010; Bentiss et al., 2000; Kokalj et al., 2010; Babic-Samardzija et al., 2005a, 2005b). The different mechanism of adsorption of inhibitor on the surface of

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metal has been proposed in terms of interaction of uncharged electron pair or pi electrons in the molecule with metal orbital (Gomez et al., 2006; Turcio-Ortega et al., 2007). Even if tremendous efforts had been made towards corrosion inhibition by inhibitor in the last several decades, the most synthetic compounds still in use were found to be expensive and toxic to the environment and human health (Wang and Jiang, 2007; Rodojcic et al., 2008). And moreover, there is increasing concern about toxicity, biodegradability and bioaccumulation of corrosion inhibitors discharge into the environment. Therefore, the study of sustainable corrosion inhibition of metal or alloy by eco-friendly inhibitor has aroused the curiosity among the corrosion scientists. The promising features of the green corrosion inhibitor are that they are renewable, biodegradable, cheap and non-toxic (Abiola and James, 2010; da Rocha et al., 2010). Therefore, they are nowadays considered as the attractive alternative for solving the problem of corrosion of metal or alloy. Many efforts in this regard were made from the last few decades. However, this development is still not enough for the practical application. There are still more needs to develop the new green corrosion inhibitors for better future, safe environment and healthy life. In this view, an attempt is made to find out a naturally occurring, cheap and environmentally safe substance that may be used as corrosion inhibitor for corrosion of mild steel in acidic medium. In this present work, we used the litchi (*Litchi chinensis*) which is shown in Fig. 1. Litchi is the sole member of the soapberry family, Sapindaceae. It is a tropical and subtropical fruit tree native to southern China, Taiwan, Bangladesh and South-East Asia. China is the main producer, followed by India. We have chosen litchi peel because litchi peel is inedible and usually discarded as a waste. We used this waste part of the litchi for prevention of corrosion of mild steel. The aqueous extract of litchi peel in 0.5 M H_2SO_4 was tested by using weight loss, potentiodynamic polarization and electrochemical impedance techniques. UV–Vis, FT-IR spectroscopy and SEM study were also used to confirm the mode of inhibition.

2. Materials and method

2.1. Mild steel

Mild steel strips having the composition (wt.%) of C (0.18), Si (0.19), Mn (0.51), P (0.044), S (0.057), Cr (0.14), Ni (0.09), Mo (0.02), Cu (0.06) and remaining Fe were used for weight loss as well as electrochemical studies. The specimens of mild steel strip were polished successively using the emery paper of 150, 180, 320, 400, 600 and 1000. The polished surface was degreased with acetone and washed with distilled water before each experiment. H_2SO4 (AR grade) solutions of 0.5 M concentration were used for all the studies.

Figure 1 Litchi fruit tree.

2.2. Preparation of extract of litchi peels

Litchi peel was dried in oven at 60 °C and grinded to powdery form. 10 g of the powder was refluxed in 100 ml double distilled water for 4 h. The refluxed solution was filtered to remove any contamination. The extract of the plant was prepared by evaporating the filtrate on water bath and drying the paste residues in desiccator. The required concentrations of solution were prepared by using the solid residues in aqueous solution of 0.5 M H_2SO_4.

2.3. Characterization of the extract of litchi peel

The extract of litchi peel was characterized by chemical tests and spectroscopic techniques. The water extract of litchi peel burned like sugar when its spot on TLC plate was heated with 4% H_2SO_4 solution. The water extract gives positive Molisch's Test, characteristic of carbohydrates and it also gives positive Fehling's test and Tollen's tests, which indicate that litchi extract contains reducing sugars. It gives negative Barfoed's test and Iodine test, confirming the absence of monosaccharide and starch in the extract. This reveals the possibility of presence of disaccharide in the extract. Further, it was characterized by UV–Vis and FT-IR spectroscopy. The solution of litchi peel extract was analysed by UV spectral measurement using shimadzu UV-1800 spectrophotometer, Japan. The absorption spectrum of the solution was determined with distilled water as reference. And FT-IR spectrum was recorded by using BX-Perkin Elmer spectra. The sample was prepared using KBr crystals and analysis was carried out by scanning the sample through a wave number range of 4000–400 cm^{-1}.

2.4. Weight loss method

In the gravimetric experiment, the weight loss study was carried out on mild steel strips of $5.0 \times 2 \times 0.025$ cm sizes at 298 K for 4 h in 0.5 M H_2SO_4 solution without and with various concentration of the plant extract. The percentage inhibition efficiency ($I\%$) of the extract was calculated from the following equation (Ostavari et al., 2009):

$$I\% = \frac{W_o - W_i}{W_o} \times 100 \quad (1)$$

where W_o and W_i refer to weight loss of strips of mild steel in absence and presence of extract of litchi peel.

2.5. Electrochemical measurements

Three electrodes system of the electrochemical cell was used for potentiodynamic polarization and electrochemical impedance studies in which mild steel was employed as working electrode which was made by coating thoroughly with epoxy resin keeping surface area of 1 cm^2 for the study. Calomel electrode

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and Platinum were used as the reference and counter electrode respectively. Before each polarization and EIS measurement, the working electrode was introduced into the test solutions and kept for 4 h to attain the open circuit potential (OCP). The electrochemical measurements were carried out under thermostatic conditions at 298 K in the range of potential from −0.7 to 0.3 V with scan rate (V/s) of 0.01 and quiet time of 2 s using computer controlled electrochemical workstation of CHI 760c model. The percentage inhibition efficiency ($I\%$) of the extract was calculated using the following equation (Abdel-Gaber et al., 2008):

$$I\% = \frac{i^{o}_{corr} - i^{i}_{corr}}{i^{o}_{corr}} \times 100 \quad (2)$$

where i^{o}_{corr} and i^{i}_{corr} refer to the corrosion current density value without and with extract, respectively, whereas in electrochemical impedance study, the measurement of the response of the electrochemical system to a.c. excitation with a frequency ranging from 10,0000 to 0.1 Hz and peak to peak a.c. amplitude of 0.005 V with quiet time of 2 s was scanned. The percentage inhibition efficiency ($I\%$) of the extract was calculated using the following equation (Znini et al., 2012):

$$I\% = \frac{R_{ct(i)} - R_{ct(a)}}{R_{ct(i,)}} \times 100 \quad (3)$$

where $R_{ct(i)}$ and $R_{ct(a)}$ are the value of the charge transfer resistance in the presence and absence of the inhibitor, respectively. The value of electrochemical double layer capacitance (C_{dl}) was calculated at the frequency, F_{max} using the following equation (Mayanglambam et al., 2011):

$$C_{dl} = \frac{1}{2\pi F_{max} R_{ct}} \quad (4)$$

where F_{max} is the frequency at which the imaginary component of the impedance is maximal.

2.6. Surface analysis

The test coupons of the size 1×1 cm^2 were dipped in 100 ml of 0.5 M H_2SO_4 solutions in the absence and presence of the extract for 5 h at 298 K. After washing the investigated coupons with double distilled water and drying the specimens, they were examined for surface analysis by Scanning electron microscope (SEM) model Leo 435 VP with an Oxford Inca energy dispersion spectrometer system.

3. Results and discussions

3.1. UV and FT-IR of extract of litchi peel

In UV spectrum, a weak absorption band at 280 nm is due to n–π^* transition in C═O of carbohydrate (as shown in Fig. 2). FT-IR spectroscopy of the extract is shown in Fig. 3. The strong absorption band at 3424 cm^{-1} is attributed to O—H stretching vibration and that at 2927 cm^{-1} is related to C—H stretching vibration. The strong band at1623 cm^{-1} is assigned to C═O stretching vibration. The C—H bending band in —CH_2 is found to be at 1407 cm^{-1}. Other absorption bands at 1138, 1102 and 1054 cm^{-1} are due to C—O stretching vibration. The absorption bands below 1000 cm^{-1} correspond to the aliphatic C—H group (Li et al., 2012a,b; Deng and Li, 2012). This result confirms that the extract contains functional groups such as (O—H) and (C═O).

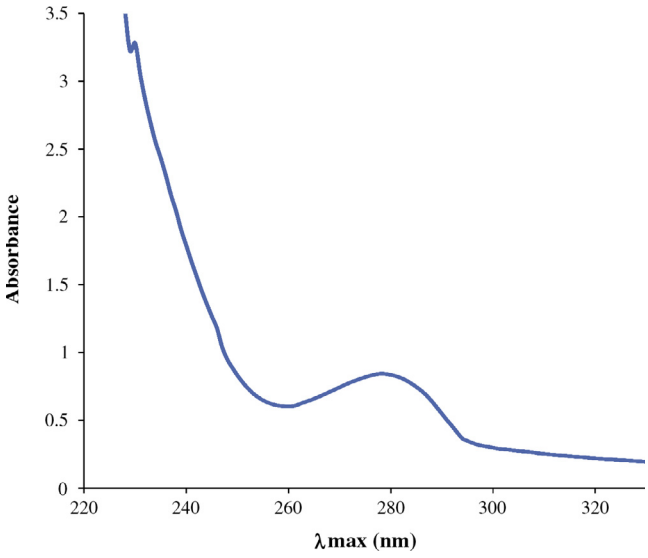

Figure 2 UV–Vis spectra of litchi peel extract (*Litchi chinensis*).

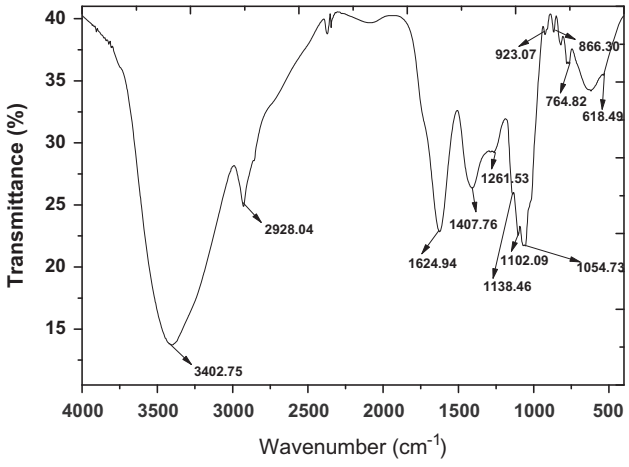

Figure 3 FTIR spectra of extract of litchi peel (*Litchi chinensis*).

3.2. Weight loss method

The percentage of inhibition efficiency ($I\%$) of extract calculated from weight loss method are listed in Table 1. The result

Table 1 The gravimetric parameters for mild steel corrosion in 0.5 M H_2SO_4 solution in the absence and presence of different litchi peel extract concentrations at 298 K.

Conc. (g/L)	Efficiency ($I\%$)
Blank	–
0.25	81.3
0.75	85.0
1.0	89.2
1.5	93.5
2.0	95.1
3.0	97.8

of Weight loss study shows that inhibition efficiency of the extract on corrosion of mild steel increases with increase in its concentration.

3.3. Potentiodynamic polarization measurement

The Tafel polarization curves for mild steel in 0.5 M H_2SO_4 solutions without and with various concentrations of extract of litchi peels at 298 K are shown in Fig. 4. The important corrosion parameters such as the corrosion current density (i_{corr}), corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (β_c and β_a) values were obtained by extrapolating the Tafel straight line on Tafel plots. And the percentage of inhibition efficiency (I%) were calculated and listed in Table 2. It is clear that the addition of the litchi peel extract to the acid solution decreases the corrosion current density (i_{corr}). This indicates that extract of litchi peel retards the rate of corrosion of mild steel. The inhibition of corrosion increases with increase in extract concentration in acidic medium. The result also reveals that the addition of the extract does not alter the values of E_{corr} significantly, indicating the mixed type nature of the extract on inhibition of corrosion on metal steel (Ibrahim et al., 2012; El-Etre, 2007; Oguzi, 2007).

From Fig. 4, it is also cleared that both cathodic reduction and anodic metal dissolution reactions were inhibited when the extracts of litchi peel were added to the acid solution. The inhibition is increased with increase in extract concentration. The values of cathodic and anodic Tafel slopes (β_c and β_a) listed in Table 2 indicate that adsorption of extract molecules modifies the mechanism of the anodic dissolution. The adsorption on anodic site may occur through interaction between metal surface and lone pair electrons of the oxygen atoms present in the extract, which decreases anodic dissolution of mild steel (Soltani et al., 2012; Abdel-Gaber et al., 2006). Whereas the values of cathodic Tafel slopes (β_c) are almost constant, it indicates that the addition of the extract to the aggressive solution does not alter the hydrogen evolution mechanism (proton reduction mechanism). This may be due to that the extract molecules are first adsorbed onto metal surface and then impede by merely blocking the active sites of the metal surface. In this way, the surface area available for proton ions is decreased, while the actual reaction mechanism remains unaffected (Khaled, 2010).

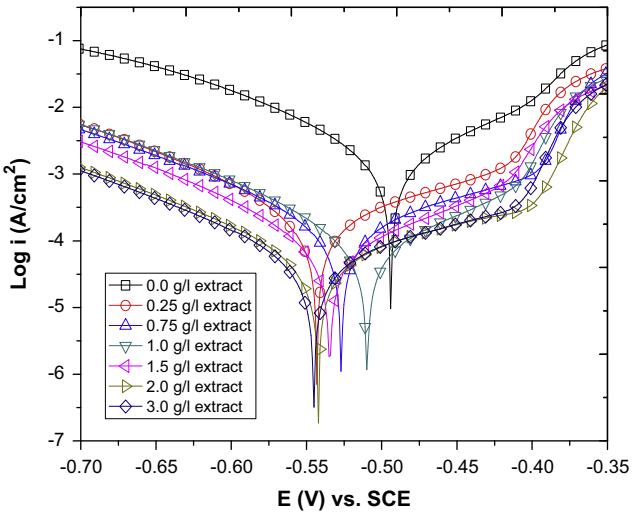

Figure 4 Potentiodynamic polarization curves for mild steel in 0.5 M H_2SO_4 solution in the absence and presence of different concentrations of the extract at 298 K.

3.4. Polarization resistance

In this study, the polarization resistance (R_p) value of mild steel in 0.5 M H_2SO_4 solution is found to increase from 13.1 Ω of the acid solution to 499.2 Ω of litchi peel extract of 0.3 g/l as listed in Table 3. The increase in R_p value suggests that the inhibition efficiency of extract on corrosion of mild steel increases with increase in its concentration (Banerjee et al., 2012; Amin and M-Ibrahim, 2011; Shukla et al., 2009).

3.5. Mechanism of inhibition

In general, the corrosion inhibitor retards the corrosion by controlling the reactions of cathode and anode by the process of adsorption of inhibitor on the solid surface of metal or alloy. It is very essential to know the nature of adsorption of the inhibitor so as to ascertain the mode of inhibition and the adsorption isotherm that fits the experimental results. Among the common adsorption isotherms such as Langmuir, Temkin and Frumkin etc., plot of the data shows that the investigated extract agreed with Langmuir isotherm which is given by the equation expressed as (El-Etre et al., 2005; Abiola and Otaigbe, 2009; Orubite and Oforka, 2004; Kalaiselvi et al., 2010; K Satapathy et al., 2009):

$$\frac{C}{\theta} = C + \frac{1}{K_{ads}} \quad (5)$$

where C is the inhibitor concentration and K_{ads} is the equilibrium constant for adsorption/desorption process of the inhibitor molecules on the metal surface.

As suggested by the plot between C/θ and C (as shown in Fig. 5) and the linear correlation coefficient of the fitted data close to 1, it confirms that the adsorption of the inhibitor molecules follows the Langmuir's adsorption isotherm.

3.6. EIS measurements

EIS measurement was conducted to study the electrode/electrolyte interface and corrosion processes that occur on metal surface in absence and presence of the extract. Nyquist plots for mild steel electrode immersed in 0.5 M H_2SO_4 solution at 298 K without and with various concentrations of the extract at the respective open circuit potential were shown in Fig. 6. It is cleared from the figure, there is a single semicircle that shows the single charge transfer process during the reaction of dissolution. The impedance data listed in the Table 4 indicate that the addition of the extract increases the values of R_{ct} and reduces the value of electrochemical double layer capacitance (C_{dl}). The increase in R_{ct} value is attributed to the formation of the protective film on the metal/solution interface (Quraishi et al., 2010; Lebrini et al., 2011; Gunasekaran and Chauhan, 2004). The decrease in C_{dl} indicates increasing in the thickness of the electric double layer (Torres et al., 2011). This result suggests that the extract molecules inhibited the corrosion of mild steel by adsorption on

Table 2 The electrochemical parameters from Tafel plot for mild steel corrosion in 0.5 M H_2SO_4 solution in the absence and presence of different litchi peel extract concentrations at 298 K.

Conc. (g/L)	$-E_{corr}$ (mV vs. SCE)	β_c (mV/Dec)	β_a (mV/Dec)	i_{corr} (mA/cm^2)	I%	θ
Blank	494	109	64	1.342	–	–
0.25	543	107	147	0.229	82.9	0.829
0.75	527	113	158	0.179	86.6	0.866
1.0	535	103	100	0.083	93.8	0.938
1.5	510	102	54	0.064	95.2	0.952
2.0	542	110	181	0.063	95.3	0.953
3.0	545	109	164	0.057	95.7	0.957

Table 3 The polarization resistance (R_p) values for mild steel corrosion in 0.5 M H_2SO_4 solution in the absence and presence of different litchi peel extract concentrations at 298 K.

Conc. (g/L)	Polarization resistance (R_p) (ohm)	I%
Blank	13.1	–
0.25	118.2	88.9
0.75	159.7	91.8
1.0	241.1	94.6
1.5	266.3	95.1
2.0	470.6	97.2
3.0	499.2	97.4

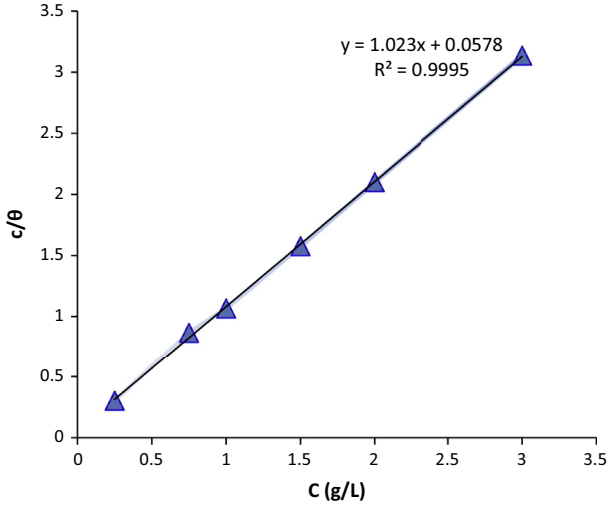

Figure 5 Langmuir adsorption isotherm for the adsorption of the extract in 0.5 M H_2SO_4 on the surface of mild steel.

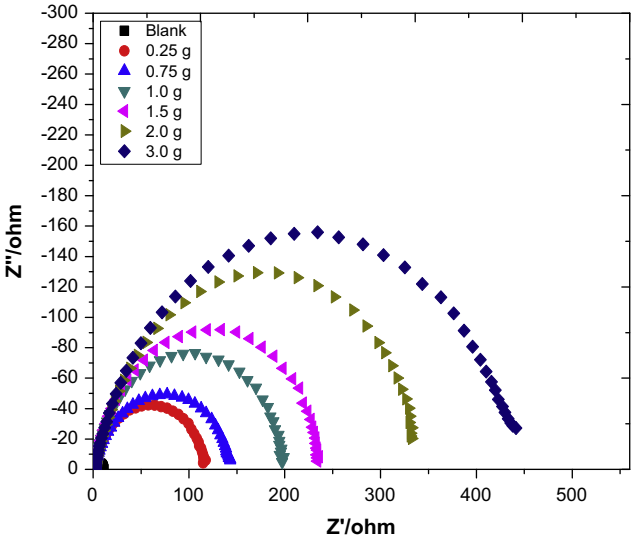

Figure 6 Nyquist plots (EIS) of mild steel immersed in 0.5 M H_2SO_4 in the absence and presence of different concentrations of the extract at 298 K.

Table 4 The electrochemical impedance parameters for mild steel corrosion in 0.5 M H_2SO_4 solution in the absence and presence of different litchi peel extract concentrations at 298 K.

Conc. (g/L)	C_{dl} (F cm^{-2})	R_{ct} (Ω cm^2)	I%
Blank	3.5×10^{-3}	10.5	–
0.25	306.5×10^{-7}	121.6	91.4
0.75	209.1×10^{-7}	154.1	93.2
1.0	101.8×10^{-7}	203.1	94.8
1.5	71.0×10^{-7}	242.5	95.7
2.0	43.9×10^{-7}	348.9	96.9
3.0	20.8×10^{-7}	486.0	97.8

the mild steel surface thereby causing the increase in R_{ct} values and decrease in C_{dl} values (Kamal and Sethuraman, 2012; Abdel-Gaber et al., 2009).

3.7. Scanning electron microscopy

In the analysis of Scanning electron microscopy (SEM), SEM micrograms of the polished surface of mild steel in 0.5 M H_2SO_4 solutions in absence and presence of the extract were shown in Fig. 7(a) and (b). In the analysis of the micrograms, there was a rough surface on iron surface which was exposed only to 0.5 M H_2SO_4 solution whereas there was a smooth surface on metal surface that was exposed to 0.5 M H_2SO_4 solution with the extract (Bothi Raja and Sethuraman, 2008; Al-Turkustani et al., 2011; Hazwan Hussin and Jain Hassin, 2011; Garai et al., 2012; Ramananda Singh, 2013). This result confirms that the extract prevents the corrosion of mild steel through adsorption of the inhibitors on metal surface. This finding supplements with the result of electrochemical techniques.

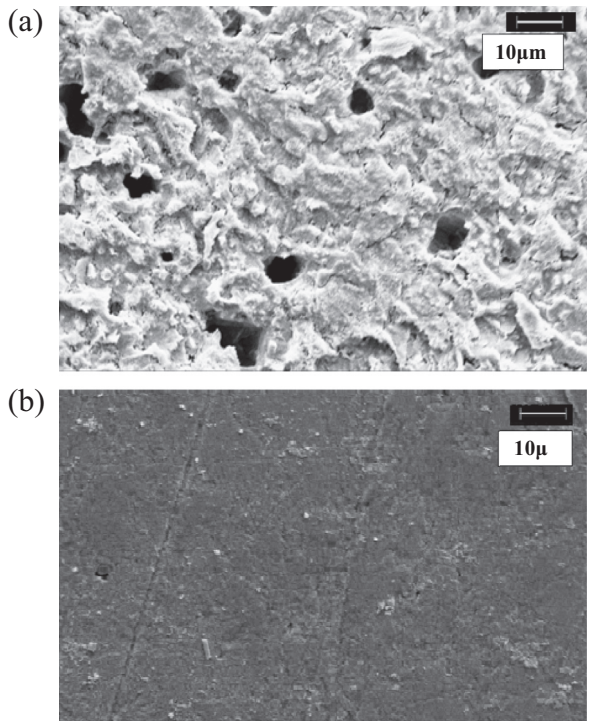

Figure 7 (a) Scanning electron micrograph of polished mild steel after exposure to 0.5 M H_2SO_4. (b) Scanning electron micrograph of polished mild steel after exposure to 0.5 M H_2SO_4 containing the extract.

4. Conclusions

The inhibition efficiency of the extract on corrosion inhibition increases with increase in concentration of extract. The result of Potentiodynamic Polarization study shows that the extract acts as mixed type inhibitor and its inhibition mechanism is adsorption. The mode of Adsorption of the extract on mild steel surface obeys Langmuir's adsorption isotherm. The EIS measurement shows that the inhibition of corrosion on mild steel is due to the formation of protective film on metal surface and the inhibition increases as the extract concentrations increase. This result is well supplemented with the results of weight loss Potentiodynamic Polarization studies. SEM study is in good agreement with the results of gravimetric and electrochemical techniques. The results of all the studies confirmed that the extract of litchi peel has potential to prevent the corrosion of mild steel in acidic environment. Therefore, the litchi peel extract is proved to be a potential green inhibitor for prevention of the corrosion of mild steel.

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