Shrimp shell waste as a potential corrosion inhibitor for mild steel in acid solution

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Abstract. Application of corrosion inhibitor is a common method to minimize corrosion reaction of steel in aqueous environments. Research for an effective eco-friendly corrosion inhibitor is required due to the toxicity issues of the most commercially available corrosion inhibitors. Many recent studies have shown that various food waste can be utilized as corrosion inhibitors. Extracting inhibitor compounds from food waste is definitely low cost, less harm, and environmentally beneficial. One example of corrosion inhibitor derived from food waste is a water-soluble chitosan (WSC) inhibitor, which can be extracted from Crustacean shells. This study used shrimp shell waste to extract WSC and test the effectiveness on inhibiting corrosion of mild steel in acid solution. The weight loss method was employed at various inhibitor concentrations and solution types. The overall findings in this study indicate that the WSC extracted from shrimp shell exhibits a good inhibition performance in 0.5 M HCl solution with the efficiency up to 94.31% at a concentration of 3 g/l at room temperature. Fourier Transform Infrared Spectroscopy and 2.2.7 UV-visible spectroscopy analysis showed that WSC molecules adsorbed on the steel surface and hinder the corrosion reactions.

1 Introduction

In oil and gas industry, the most preferred protection against corrosion are inhibitors[1]. Inhibitors are chemical compounds added to an environment in small concentration to minimize the corrosion reaction. However, most of the commercially available inhibitor compounds are toxic. Efforts to find sustainable natural compounds to be employed as eco-friendly corrosion inhibitors, which called as green inhibitors, have been increased in recent years. Research and development of green inhibitors have been performed using plant extract, vegetable oil, juice, or waste [2]. Most of the researches are done by using plant extracts. Therefore, further research using other resources is still required, for example, biopolymers. Biopolymers can be utilized as corrosion inhibitors due to their high adhesion effectiveness, big molecule size, and large adsorption capability[3]. One of biopolymers that can be used as corrosion inhibitor is chitosan and its derivative substances[3,4].

One source of chitosan that have a potential to be developed as green inhibitor is shrimp shell waste. Indonesia is known as a maritime country because the area of the ocean in Indonesia reaches two-thirds of its total area. Based on data reported by the Ministry of Marine Affairs and Fisheries in 2020, shrimp is one of the largest export commodities with a volume of 239.28 million kilograms and a value of 2.04 billion rupiah[5]. Shrimp farming products in Indonesia reached 911.2 thousand tons in 2020, and is targeted to reach two million tons by 2024[6]. The export of frozen shrimp causes environmental problems, one of which is the accumulation of carapace and peel waste. Until now, these by-products have been utilized as raw materials for the cracker industry, *petis*, shrimp paste, broth, and animal feed ingredients. However, this utilization only accounts for 30% of the total waste[7]. Besides being cheap and easily available, the utilization of shrimp shell waste as a source of chitosan that can be used for corrosion inhibitors is a right step in the utilization of waste so as to reduce the volume of organic waste.

Shrimp shell waste contains chitin compounds as a source of chitosan, which is economically valuable and widely used for industrial purposes. The chitin content in shrimp shell waste is about 20-50% by dry weight[7]. Chitin as a raw material for chitosan is an acetylamine polysaccharide that is biodegradable and non-toxic. Chitosan $(\mathrm{C_6H_{11}NO_4})_n$ is a compound that can be synthesized from chitin in the form of an amorphous solid that is yellowish-white in color, polyelectrolyte, and has groups of $-\mathrm{OH}$ or $-\mathrm{NH_2}$ that can bind to Fe atoms in low carbon steels [2]. Chitosan can be a corrosion inhibitor compound because the amine and hydroxyl groups have free electron pairs on oxygen and nitrogen atoms so that they can be well adsorbed on metal surfaces. However, chitosan is insoluble in water and has a low solubility in neutral media (pH > 6) due to its large molecular weight, so that the use of chitosan in aqueous solutions is limited[2]. Therefore, the conversion of chitosan into water soluble chitosan (WSC) through the addition of hydroxyl ($\mathrm{OH^-}$)

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compounds was carried out[8]. The synthesis of WSC was carried out through a series of demineralization, deproteination, deacetylation, and conversion of chitosan into WSC[3,8]. Some previous studies on green inhibitors extracted from seafood waste as a source of chitin can be seen in Table 1. Research on the adsorption ability of inhibitors currently focuses more on the effects of metal composition and inhibitor molecules[9]. In this research, the effect of WSC concentration and type of solution on the efficiency of green inhibitors synthesized from shrimp shell waste on the corrosion of carbon steel was investigated.

2 Materials and Methods

2.1 Material

The chemical composition of ASTM A36 steel sample based on Optical Emission Spectrometer (OES) test is displayed in Table 2. Shrimp shell waste was obtained from a seafood restaurant in Bandung, Indonesia.

2.2 Methods

2.2.1 Extraction of water soluble chitosan (WSC)

The WSC extraction process was carried out through four main stages, namely demineralization using 7% HCl, deproteination using 3.5% NaOH, deacetylation using 60% NaOH, and conversion of chitosan into WSC using 2% CH_3COOH and 6% H_2O_2 [2,8].

Firstly, the shrimp shells were washed using clean water, then dried under the sun for five days to make sure they were completely dry. Afterwards, the dry shrimp shells were ground using food chopper to achieve a 60-mesh sieve particle size. A total of 100 grams of dry shrimp shells were weighed and used as raw material for the WSC extraction process according to the flow chart shown in Fig. 1.

2.2.2 Preparation of test solution

The solutions used were 0.5 M HCl and 0.5 M H_2SO_4. HCl pro-analysis 37% (Merck) and H_2SO_4 pro-analysis 98% (Smart-Lab) were diluted in distilled water to get the concentration of 0.5 M based on Volumetric Law ($M_1 \times V_1 = M_2 \times V_2$). The WSC inhibitors that have been extracted from shrimp shells were added to the test solution and stirred using a magnetic stirrer. The test solution without the addition of WSC is called as blank solution.

2.2.3 Immersion test

The method of measuring the corrosion rate through the immersion test was carried out in accordance with the ASTM G31 Standard Practice for Laboratory Immersion Corrosion Testing of Metals. Carbon steel samples with dimensions of 50 x 20 x 8 mm were polished with SiC paper grit 60 to 2000, consecutively. Prior to immersion tests, the samples are subjected to room temperature ultrasonic cleaning in ethanol 96% for five minutes and followed by the air-drying step. Each sample was weighed using analytical balance before the test.

The immersion tests were conducted in a closed beaker glass for 24 h. After 24 hours, the samples were taken out and corrosion product formed on the surface was removed by immersion in a 50 g/l HCl solution for 6 minutes, and then rinsed with distilled water. The clean sample was then weighed again. The corrosion rate as well as the inhibitor efficiency value then calculated by the following equation:

$$\mathrm{CR} = \frac{\Delta \mathrm{W} \times \mathrm{K}}{\rho \times \mathrm{A} \times \mathrm{t}} \tag{1}$$

$$\%\mathrm{IE} = \frac{\mathrm{CR_{blank}} - \mathrm{CR_{inh}}}{\mathrm{CR_{blank}}} \times 100\% \tag{2}$$

where *W* is the weight loss to corrosion in grams; *K* is the constant used (for mm/year, the value of K = 87,600); *ρ* is the density of the steel in gr/cm^3; *A* is the immersed surface area in cm^2, *t* is the immersion time in hours, *%IE* is the inhibitor efficiency, CR_{blank} and CR_{inh} are the corrosion rates without and with inhibitor added, respectively. All immersion test were conducted in duplicate.

2.2.4 Electrochemical test

Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization (PDP) were carried out on a 10 x 10 mm sample using a potentiostat (Corrtest CS350). Resin-mounted steel sample was used as a working electrode, Ag/AgCl as a reference electrode, and graphite as a counter electrode. Firstly, Open circuit potential (OCP) was measured after conditioning time, followed by EIS test at a frequency range from10,000 Hz to 0.1 Hz at OCP using an AC current with 10 mV of amplitude. The equivalent electrical circuit model was determined using *ZView* software. The PDP test was conducted with a scan rate of 0.167 mV/s at an initial potential of -300 mV to +300 mV against the OCP.

2.2.5 Surface Characterization

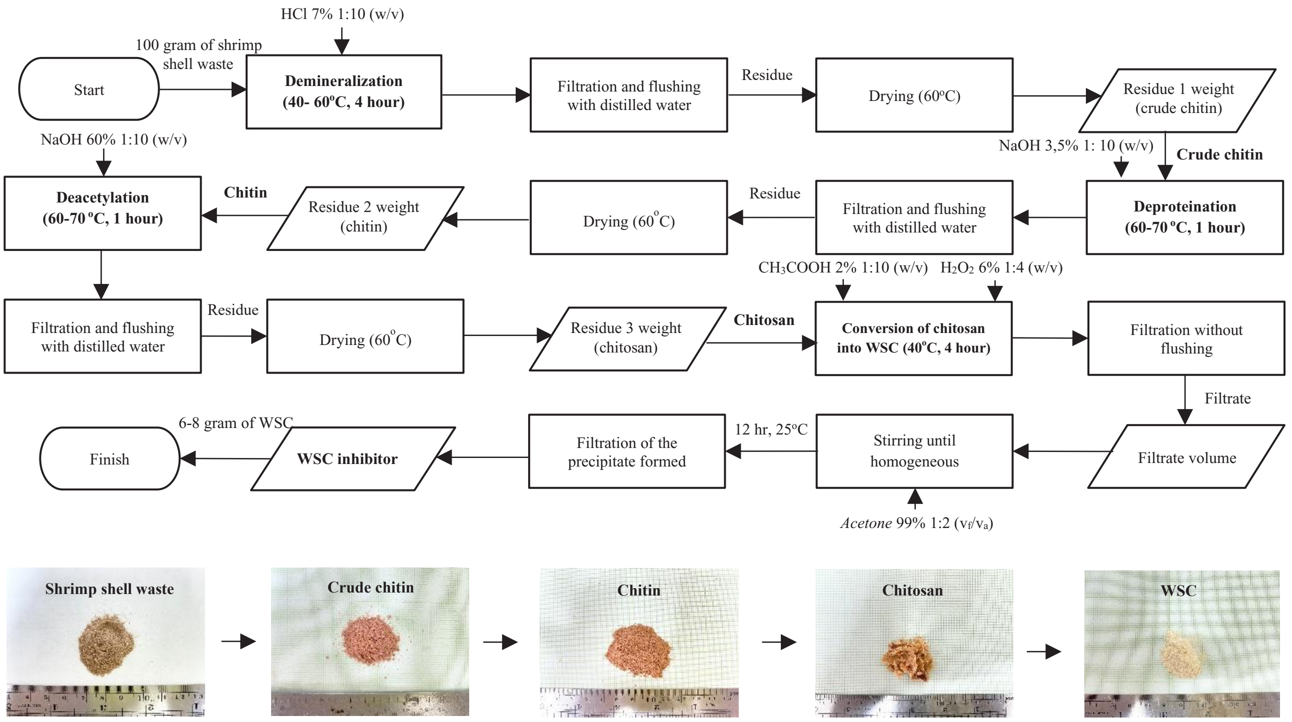
Scanning electron microscope (SEM) analysis were conducted on the steel surface after 24 hours immersion test in the blank solution and the WSC-added solution.

Table 1. Published research of shell waste extract used as green inhibitor

Sources	Sample	Optimum Concentration	Electrolyte Solution	T (°C)	Corrosion Rate Measurement Method	IE (%)	Ref
Shrimp shell waste	Carbon steel SS400	200 ppm	HCl 1 M	28	Immersion test	74.6	[8]
Shrimp shell waste	Tin plate	1300 ppm	NaCl 2%	25	Immersion test	72.7	[3]
				25	PDP	91.4	
Mussel shell waste	Tin plate	1300 ppm	NaCl 2%	25	Immersion test	54.5	[3]
Shrimp shell waste	Copper	8×10^{-6} M	HCl 0,5 M	25	Immersion test	93	[10]
Shrimp shell waste	Low carbon steel	100 ppm	HCl 1 M	25	PDP	73.5	[11]
Shrimp shell waste	Carbon steel	5×10^{-3} M	HCl 1 M	25	PDP	95.1	[12]
Crab shell waste	Carbon steel	5×10^{-3} M	H_3PO_4 2 M	25	Immersion test (12 hours)	94.5	[13]

Table 2. Chemical composition of ASTM A36 steel

Element	Fe	C	Si	P	Mn	Ni	Cu	S
wt.%	99.0257	0.19581	0.21061	0.01321	0.47603	0.00540	0.00872	0.01280
Element	W	Ti	Sn	Al	Cr	Mo	V	Zn
wt.%	0.00052	0.00187	0.00060	0.00316	0.01156	0.01018	0.00004	0.00035



Light brown, fine Dark brown, fine Dark brown, fine Golden brown, sticky Yellowish white, fine

Fig. 1. WSC extraction procedure[2,8]

2.2.6 FTIR spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) analysis were conducted on the dried WSC powder and the scraped corrosion products from steel surface after the immersion test to ensure the adsorption of WSC molecules on the steel surface.

2.2.7 UV-visible spectroscopy

UV-visible spectroscopy was conducted in the wavelength range of 190 to 1100 nm on the WSC-added solution before and after the 24 hours immersion test.

3 Results and Discussion

3.1 Immersion test analysis

3.1.1 The effect of WSC concentration on corrosion rate

Immersion tests were conducted in blank HCl solution and WSC-added HCl solution with WSC concentration varied from 1 to 4 g/L. Fig. 2 shows the effect of WSC concentration on corrosion rate and inhibitor efficiency in 0.5 M HCl solution. From Fig. 2, the highest inhibition efficiency up to 94.31% is obtained when 3 g/L of WSC was added. Based on the inhibitor efficiency value, the degree of surface coverage (θ) can also be determined. The greater value of surface coverage indicates the wider the surface area protected by the inhibitor molecules so that the corrosion reaction is inhibited [14]. The degree of surface coverage can be determined by the following equation[15].

$$\theta = \frac{\text{IE }(\%)}{100} \tag{3}$$

If the concentration of inhibitor added to the solution exceeds the optimum dose, the activity of the inhibitor will decrease slightly due to the desorption of some molecules back into the solution[14]. Desorption results in a lower surface coverage, which promotes increase in corrosion rate. As seen in Fig. 2, addition of 4 g/L WSC gives a slightly higher corrosion rate than 3 g/L, which indicates that 3 g/L is the optimum WSC concentration.

3.1.2 The effect of solution types on corrosion rate

For comparison, the immersion tests were also carried out in 0.5 M H_2SO_4 solution and the results are shown in Fig. 3. At the same WSC concentration, corrosion rate was immensely higher in 0.5 M H_2SO_4 compared to 0.5 M HCl. WSC inhibitor shows higher efficiency in 0.5 M HCl than that in 0.5 M H_2SO_4. Note that corrosion rate of steel sample in 0.5 M H_2SO_4 blank was almost double than the corrosion rate in 0.5 M HCl blank. The higher concentration of H^+ ions in the H_2SO_4 solution is thought to have an influence on the reduction reaction in the system so that the corrosion is faster.

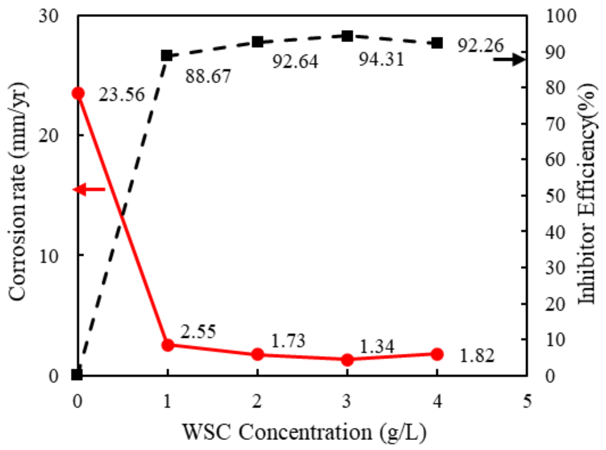


Fig. 2. Effect of inhibitor concentration on corrosion rate and inhibitor efficiency in 0.5 M HCl solution from 24 hours immersion test

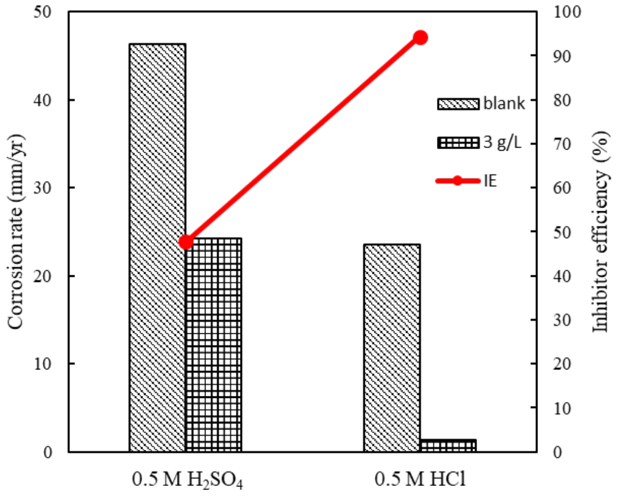


Fig. 3. Corrosion rate and inhibitor efficiency in 0.5 M H_2SO_4 and 0.5 M HCl solutions

3.2 Electrochemical test analysis

3.2.1 Open circuit potential analysis

OCP is the potential value in a corrosion cell when no current has been applied[16]. The OCP value is a description of the corrosion potential value when the system reaches steady state condition. OCP measurements were recorded after conditioning time, when the potential has reached a stable value. Fig. 4 shows the OCP for 100 seconds for steel sample in 0.5 M HCl solution with and without WSC addition.

It can be seen that the OCP was slightly increased with the WSC addition. The small difference (< 85 mV) in OCP value indicates that the presence of the inhibitor does not greatly affect the potential in the system, which indicates that the WSC is performed as a mixed type inhibitor[17,18]. This type of inhibitor is expected to trigger and accelerate a protective passive layer formation on the metal surface, which inhibits and blocks the sites for anodic and cathodic reactions[17,18].

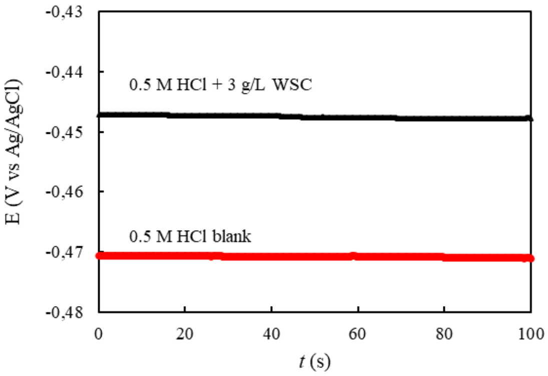


Fig. 4. OCP test results of steel sample in 0.5 M HCl solution with and without WSC addition

3.2.2 EIS analysis

Nyquist and Bode plots from EIS test of 0.5 M HCl blank solution and with addition of 3 g/L WSC are plotted in Fig. 5. Based on the characteristics of the Nyquist plot, the semicircular shape indicates the presence of a constant phase element (CPE) paralleled with a resistor[19]. CPE is used to indicate the possibility of non-ideal capacitance expressed by α value[20]. If the value of $\alpha = 1$, then it indicates that the impedance value of the CPE is equal to the impedance value of the capacitor. If the value of $\alpha = 0.5$, then the CPE impedance value is equal to the Warburg impedance. However, if the value of $\alpha = 0$, then the CPE impedance value is equal to the resistor impedance[20]. When the value of α is getting closer to 1, it indicates that the properties of the passive layer formed on electrode surface is getting better[20]. However, it is generally rare to find α value that shows an absolute 1 or reflects the ideal capacitor impedance value[21]. This is due to the uneven surface of the working electrode[22]. In this study, α_{dl} obtained was around 0.9.

The equivalent electrical circuit models obtained from curve fitting are illustrated in Fig. 6. The fitted models for steel in HCl blank solution and WSC-added solution were not identical. In both models, Rs is the solution resistance, Rp is the polarization resistance or known as charge transfer resistance, CPEdl is the constant phase element on the double layer, and CPEi is the constant phase element on the adsorbed inhibitor film. Rs-(CPEdl/Rp) circuit in Fig. 6 (a) indicates that the steel is actively corroded. Meanwhile, Rs-(CPEi(Ri(CPEdl/Rp))) circuit illustrates the presence of an adsorbed inhibitor film on the steel surface. However, this layer is porous, allowing penetration of the solution to the electrode surface. The value of each component in the equivalent circuits are presented in Table 3.

It can be seen in Nyquist plot that the addition of WSC produce a larger curve diameter, which means that the WSC inhibitor increased the steel corrosion resistance proved by a higher polarization resistance (R_p). Inhibitor efficiency can be determined using the following equation[22]:

$$\%\text{IE} = \frac{Rp_{inh} - Rp_{blank}}{Rp_{inh}} \text{ x } 100\% \tag{4}$$

where Rp_{inh} is the polarization resistance in WSC-added solution and Rp_{blank} is the polarization resistance in blank solution. The inhibitor efficiency calculated from equation (4) was 82.63%. Thus, it can be concluded that based on EIS analysis, WSC inhibitor was successfully improved the steel corrosion resistance in HCl solution.

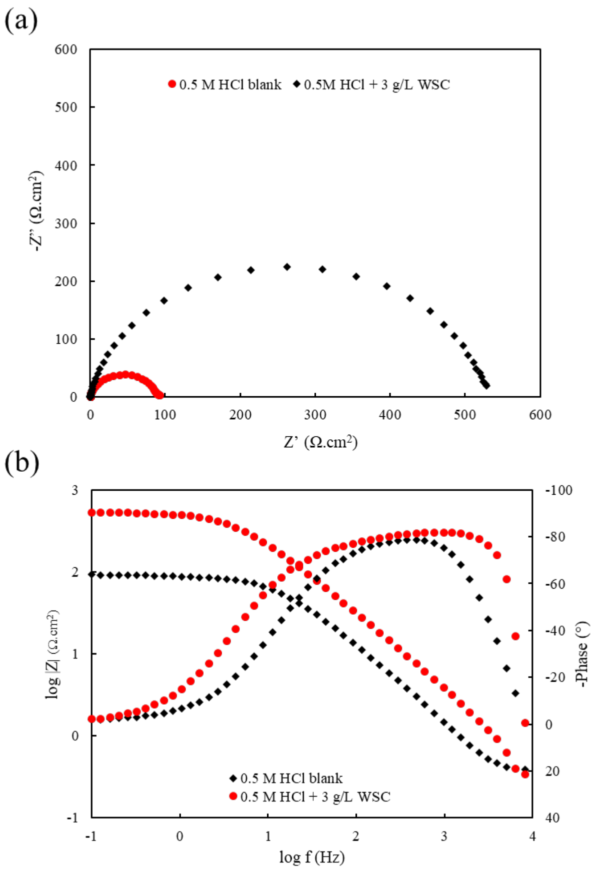


Fig. 5. (a) Nyquist and (b) Bode plot from the EIS test results of steel sample in 0.5 M HCl solution with and without WSC addition.

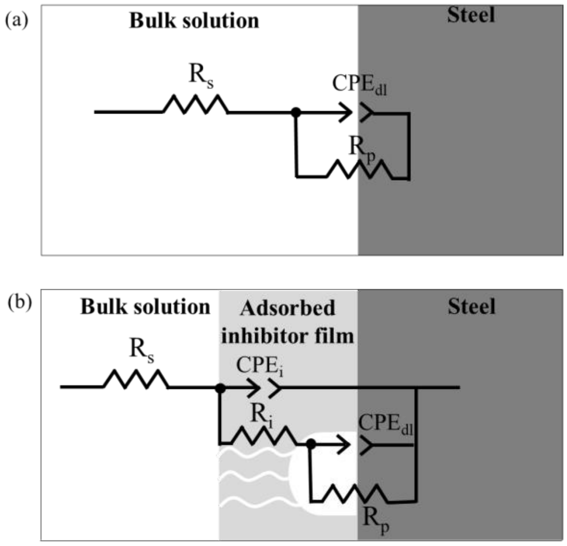


Fig. 6. Equivalent circuit models for (a) 0.5 M HCl blank and (b) WSC-added solution

3.2.3 PDP analysis

PDP tests were conducted on steel samples immersed in HCl blank solution and with the addition of 3 g/L of WSC. The PDP curves are shown in Fig. 7. Tafel extrapolation was performed on each polarization curve to obtain polarization parameter data and the WSC inhibition efficiency, as shown in Table 4. Corrosion rate then can be measured by equation below[23,24]:

$$CR = \frac{K_1 . i_{corr} . EW}{\rho} \qquad (5)$$

where CR is the corrosion rate (mm/year), K_1 is a constant (3,27 x 10^{-3} mm.g/µ.A.cm.yr), ρ is the density of the steel sample (g/cm^3), and EW is the equivalent weight of the steel sample, which can be calculated using procedure in ASTM G102.

The obtained corrosion potential (E_{corr}) values for both HCl blank solution and WSC-added solution were almost similar, while the corrosion current density (i_{corr}) in WSC-added solution was slightly lower than that of the HCl blank solution. The anodic (βa)and cathodic (βc) Tafel slopes can be associated with the dissolution of Fe metal at the steel surface and hydrogen gas formation at the cathodic area, respectively [8]. Based on the slight shift of the E_{corr} value and the change of βa and βc value, it can be assumed that the WSC inhibitor is a mixed-type inhibitor that protects both the anodic and the cathodic area on the steel surface, hinder both anodic and cathodic reactions[17,18].

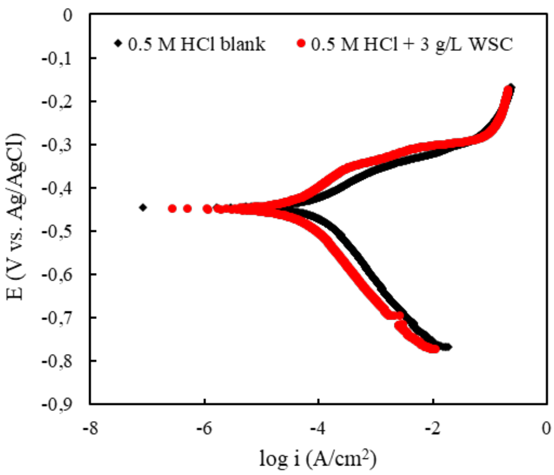


Fig. 7. PDP test results of steel sample in 0.5 M HCl solution with and without WSC addition

Table 3. EIS parameters for steel sample in 0.5 M HCl blank and WSC-added solutions

Solution	R_s (Ω.cm^2)	R_i (Ω.cm^2)	R_p (Ω.cm^2)	CPE_i (µF.cm^2)	α_i	CPE_{dl} (µF.cm^2)	α_{dl}	χ^2	%IE
0.5 M HCl Blank	0.377	-	90.01	-	-	164	0.969	0.015	-
0.5 M HCl + WSC 3 g/l	0.710	0.411	519.50	3.91x10^{-3}	1.910	91	0.914	0.011	82.63

Table 4. Polarization parameters for steel sample in 0.5 M HCl blank and WSC-added solutions

Solution	β_a (V/dec)	β_c (V/dec)	E_{corr} (V vs Ag/AgCl)	i_{corr} (µA/cm^2)	Corrosion rate (mm/yr)	%IE
0.5 M HCl Blank	0.074	0.156	-0,445	85.88	0.981	-
0.5 M HCl + WSC 3 g/l	0.089	0.141	-0,449	36.40	0.416	57.61

3.3 Characterization analysis

3.3.1 SEM analysis

Fig.8 shows the secondary electron (SE) images of the surface morphology of samples after the immersion tests in the absence (Fig.8a) and presence (Fig.8b) of 3 g/L WSC inhibitor. In HCl blank solution, massive pits were formed, which implies the corrosion has severely occurred on the steel surface. On the other hand, in WSC-added solution, the steel surface remains intact with clean surface without any pits. Hence, it is clear that the addition of WSC was significantly reduce the steel corrosion in HCl solution.

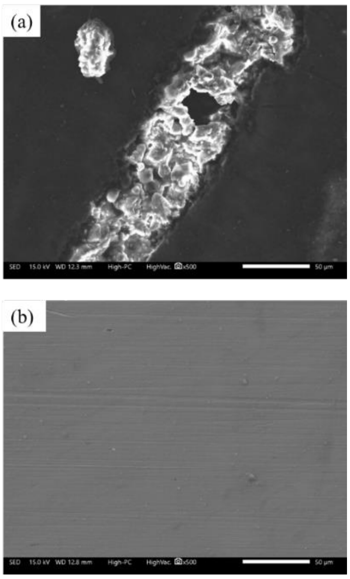


Fig. 8. SEM images of steel sample after immersion in (a) 0.5 M HCl blank and (b) WSC-added solution

3.3.2 FTIR analysis

FTIR analysis is used to analyze the functional groups in an organic compound. Here, FTIR was used to determine the chemical bonds present in the WSC obtained from the series of extraction procedure, and compare the result with that of the corrosion product on the steel surface formed during the immersion test. As seen in Fig. 9, both spectrum shows comparable peaks. The peaks at 3410.15 cm^{-1} show O-H group vibration, 2987.09 cm^{-1} show C-H sp^3 group vibration, 2468.88 cm^{-1} show C ≡ N group vibration, 1751.36 cm^{-1} show C=O group vibration, 1643.35 dan 1446.61 show N-H amide group vibration, 1068.56cm^{-1} and 1029.99 cm^{-1} show C-O-C group vibration, and many peaks between 866.04 cm^{-1} to 516.92 cm^{-1} show NH_2 and NH bending group vibration. These identified bonds are in accordance with FTIR spectrum of WSC extract that have been carried out by Harmami et al. [3] and Amanah et al. [9].

The presence of similar groups between WSC and corrosion product convince that the WSC molecules were really adsorbed onto the steel surface and protect the steel from the aggressive ions in the solution. The transmittance value of the corrosion product is greater than that of the WSC because the WSC molecule is an intact molecule of a long chemical bond chain so that only a small amount of light can be transmitted. Meanwhile, in the corrosion product only some portion of the WSC compound adsorbed on the steel surface so that more light can be transmitted by the molecule.

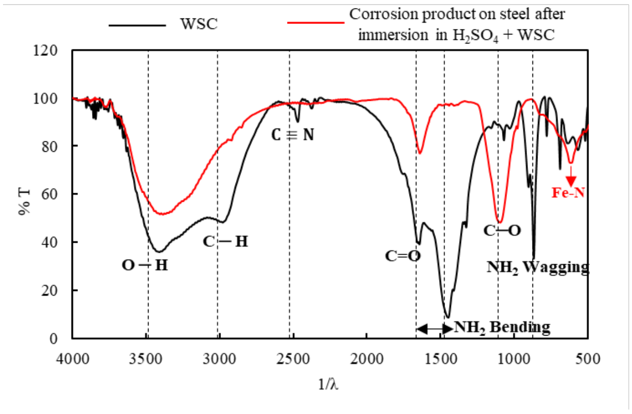


Fig. 9. FTIR spectrum of WSC extracted from shrimp shell compared with corrosion products formed on steel surface after immersion in WSC-added H_2SO_4 solution

3.3.3 UV-vis analysis

UV-vis analysis aims to prove that there were organic compounds adsorbed onto the steel surface. In Fig. 10, the comparison of the absorbance value of the WSC-added H_2SO_4 and WSC-added HCl solutions before and after immersion test are displayed. In the UV-Vis results of the WSC-added H_2SO_4 solution, there are absorbance peaks at the same wavelength of 203 nm and 399 nm in both before and after immersion. Both peaks indicate the occurrence of electron transition π-π* and n-π*. Similarly, in WSC-added HCl solution, there are absorbance peaks at the same wavelength of 206 nm and 485 nm for both before and after immersion, which indicates the occurrence of electron transitions π-π* and n-π*. Electron transition π-π* occurs in functional groups that have double bonds, while n-π* occurs on functional groups with single bonds[25].

It also can be seen that the solution before immersion shows higher peaks than that of after immersion. In WSC-added H_2SO_4 solution, the absorbance value at 203 nm for the solution before immersion was 2.5, while after immersion it drops to 1.9. Then at 399 nm, the absorbance value of the solution before immersion was 0.04, while after immersion it drops to 0.02. This is because in the solution before immersion, higher amount of organic compounds presence in the solution, which absorb more lights. Meanwhile, after immersion some organic compounds have been adsorbed to the steel surface so that the absorbance is lower. Therefore, UV-vis test results also confirmed that the WSC molecules were really adsorbed on the steel surface during immersion.

In addition, the UV-vis results of the H_2SO_4 solution showed that in the wavelength range of 290 nm - 460 nm, the absorbance value of the solution after immersion became higher than that of before immersion. The increase of absorbance at low wavelengths and the decrease of absorbance at high wavelengths can be caused by the formation of inter-molecular complexes that occur in the solution[29]. In this experiment, it is thought to be caused by the formation of complexes between Fe^{2+} ions with organic compounds contained in WSC. Thus, the UV-Vis results can be an indication that the WSC extract is adsorbed on the steel surface and is able to reduce the dissolution of Fe.

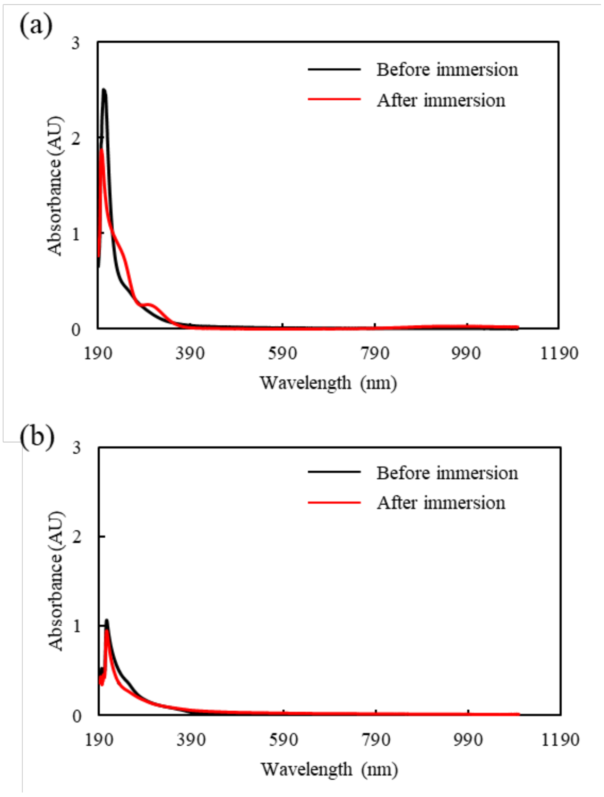


Fig. 10. UV-vis spectrum of (a) WSC-added H_2SO_4 and (b)WSC-added HCl solutions before and after immersion of steel sample

4 Conclusions

The WSC extracted from shrimp shell waste was proved to have a good inhibition performance for steel in acid solutions. Based on the immersion test, the optimum concentration of WSC in 0.5 M HCl solution was 3 g/L with the inhibition efficiency of 94.31%. Electrochemical tests and characterization analysis also confirmed that the WSC reduce corrosion tendency by adsorbed on the steel surface and hinder the corrosion reactions. SEM images showed a clean and intact surface after immersion in WSC-added HCl solution. Thus, it is confirmed that shrimp shell waste has a potential to be further developed as a green corrosion inhibitor.

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