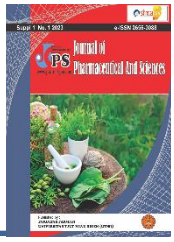
JOURNAL OF PHARMACEUTICAL AND SCIENCES
Electronic ISSN: 2656-3088
Homepage: https://www.journal-jps.com

ORIGINAL ARTICEL

JPS |Suppl 1 | No. 1 | 2023 |pp.168-176



Utilization of tannin extract from spent coffee ground (SCG) as a green corrosion inhibitor on ferrous metals in acidic and alkaline media

Pemanfaatan ekstrak tanin dari ampas kopi sebagai inhibitor korosi ramah lingkungan pada logam besi dalam media asam dan basa

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ABSTRACT
The inhibitory effect of spent coffee grounds (SCG) tannin extract as a corrosion inhibitor on ferrous metal in acid and alkaline media has been carried out. The results of the evaluation of the corrosion rate (CR) and inhibition efficiency (IE) in this study used the weight loss method. The Internasional Standard (SI) unit for calculating the corrosion rate was mm/year (mmpy). The results of the characterization of SCG tannin extract using Fourier Transform Infrared Spectroscopy (FTIR) showed that the functional groups were O-H, aliphatic C-H, C=O ester, aromatic C=C, C-O-H, and C-O-C ether which indicated the presence of tannin compounds in SCG. The results of the research showed that the maximum concentration of SCG tannin extract in inhibiting corrosion in an acidic medium was 1000 ppm with an average CR of 2.2520 mmpy and an IE of 29.23%, while in the alkaline medium, it was 1000 ppm with an average CR 0.0592 mmpy and IE 50.93% for five days of soaking time, respectively. The surface morphology of the corroded ferrous metals was monitored with a USB digital microscope at 1000x magnification.

Keywords: *Tannin extract; Spent coffee grounds; Corrosion inhibitor; Ferrous metal; Acidic and alkaline media*

ABSTRAK
Pengujian efek penghambatan ekstrak tanin dari ampas kopi sebagai inhibitor korosi pada logam besi dalam larutan asam dan basa telah berhasil dilakukan. Hasil pengujian laju korosi pada penelitian ini menggunakan metode kehilangan massa. Satuan Standar Internasional (SI) yang umum digunakan untuk perhitungan laju korosi adalah mm/year (mmpy). Hasil karakterisasi ekstrak tanin daril ampas kopi menggunakan *Fourier Transform Infrared Spectroscopy* (FTIR) diperoleh gugus fungsi, yaitu O-H, C-H alifatik, C=O ester, C=C aromatik, C-O-H, dan C-O-C eter yang menunjukkan bahwa adanya senyawa tanin di dalam ampas kopi. Hasil penelitian menunjukkan konsentrasi maksimum ekstrak tanin metanol ampas kopi dalam menginhibisi korosi dalam larutan asam yaitu 1000 ppm dengan nilai rata-rata laju korosi 2,2520 mmpy dan efisiensi inhibisi sebesar 29,23%, sedangkan dalam larutan basa yaitu 1000 ppm dengan nilai rata-rata laju korosi 0,0592 mmpy dan efisiensi inhibisi sebesar 50.93 % selama 5 hari waktu perendaman. Morfologi permukaan logam besi terkorosi dimonitor dengan menggunakan *USB digital microscope* pada perbesaran 1000x.

Kata kunci*: Ekstrak tanin; Ampas kopi; Inhibitor korosi; Logam besi; Media asam dan basa*

INTRODUCTION

Coffee drinks are the most popular in the world and are consumed by various groups. Indonesia is one of the largest coffee producers in the world. Data shows that Indonesia exports coffee to various countries worth US$ 588,329,553.00 annually. Based on the results of the national socio-economic survey (SUSENAS) conducted by Badan Pusat Statistik (BPS), it is known that in 2016, coffee consumption per capita was 0.871 kg/capita/year (Pusat et al., 2016). Increased production of coffee drinks causes an increase in spent coffee grounds (SCG) (Susilayati et al., 2022). The content of SCG includes total carbon 47.8-58.9%, total nitrogen 1.9-2.3%, ash 0.43-1.6%, and cellulose 8.6% (Oko et al., 2021). SCGs are usually thrown away after use, which has led researchers to begin studying the utilization of SCGs. One can be used as activated charcoal as an adsorbent (Purwanto & Diasmara, 2020). SCG is rich in fiber, protein, essential amino acids, and sugar (Bouhlal et al., 2020). The chemical components contained in SCG are caffeine, chlorogenic acid, trigonelline, lipids, and tannins (Chismirina et al., 2014).

Tannins are a complex mixture of non-toxic and biodegradable polyphenolic compounds extracted from plants. Tannins that come from plants mainly consist of flavonoid polymers and are phenolic compounds. Biologically, the phenolic compounds contained in tannins can act as metal chelators (ligands). Tannins have an OH- group in the ortho position on the aromatic ring to form chelates with iron and other metal cations (for example, copper) (Nardeli et al., 2019).

Iron is a metal that corrodes quickly; therefore, some effort is needed to inhibit this process. A corrosion inhibitor is one of the most effective steps to inhibit corrosion (Ludiana & Handani, 2012). According to their essential ingredients, Corrosion inhibitors are divided into inhibitors from organic compounds and inorganic compounds. Organic inhibitors generally come from extracts of natural materials containing N, O, P, and S atoms and atoms with lone pairs of electrons. Today's inorganic inhibitors are sodium nitrite, chromate, phosphate, and zinc salts. Nowadays, corrosion inhibitors from organic compounds are preferred because they are safer and do not contain toxins. Its low cost and abundance also support this and its good corrosion resistance performance (Pratiwi et al., 2020).

Haryono et al. (2010) have researched the utilization of natural extracts such as pine resin, tobacco, coffee, and gambier as iron corrosion inhibitors in seawater using the weight loss method. The research results showed that pine resin extract was the best inhibitor for reducing iron corrosion in seawater environments, with a reduction in the corrosion rate of 87.22% in the temperature range of 29-37°C. Meanwhile, for tobacco, coffee, and gambier extracts, the reduction in corrosion rate was 63.75%, 57.84%, and 11.34%, respectively.

Another study was conducted by Shahadad and Hussin (2020), who used tannin extract from mangrove bark as a Zn-P insulator for anticorrosive of mild steel in a 3.5% NaCl solution. The research showed that tannin extract improved corrosion inhibition performance with optimum inhibitory potential found at 4 g L^{-1} of a tannin concentration. Research on the use of tamarind peel tannin extract (TSTE) was also studied by Abdulmajid et al. (2019) to inhibit the corrosion of mild steel in a 0.5 M HCl solution using methanol and acetone solvents. The TSTE concentration increased the inhibition efficiency to a maximum concentration of 1000 ppm. The inhibition efficiency was 87.77% and 86.34% for methanol extract (TME) and acetone extract (TAE), respectively.

The inhibitive effect of bay leaf tannin extract as a corrosion inhibitor on aluminum plates in a 0.5 M H2SO4 medium was also conducted by Harianja & Heltina (2020), which resulted in an inhibition efficiency of 70.37% with a corrosion rate of 1.105 mmpy. This natural compound has proven its ability to be an eco-friendly corrosion inhibitor on several metals and their alloys in various aggressive media. Based on the overview above, this research aimed to determine the corrosion rate (CR) and inhibition efficiency (IE) of utilizing tannin extract in methanol solvent from SCG waste coming from Bener Meriah, Aceh, as a corrosion inhibitor in different concentrations of iron plates in acidic and alkaline media with a concentration of 0.5 M. Weight loss methods was applied in this study.

MATERIALS AND METHODS

Materials

The materials used in this research were an Iron plate (dimension 7.5 cm x 4 cm x 0.1 cm), Methanol (CH_3OH), Acetone (CH_3COCH_3), Sulphuric acid (H_2SO_4), Sodium Hydroxide (NaOH), Tannin commercial, and distilled water (H_2O). Merck purchased all the solvents. SCG wastes were obtained from coffee shops around Bener Meriah, Central Aceh.

The equipment used in this study includes various appliance glasses, glass plates, pipettes, analytical balance, rotary evaporators, desiccators, shakers, ovens, and other supporting equipment. Meanwhile, the instruments used were FTIR and a digital USB microscope.

Preparation of tannins extracts from SCG waste.

The SCG wastes were dried at room temperature, reducing the size of the waste to granule form by using a grinding machine, and then the granule waste was sifted using a 30 mesh sieve. 10 g of granule SCG was extracted with 100 mL of methanol using a wise shaker at 145 rpm for 24 hours at ambient temperature. The filtrate was collected via vacuum filtration. The collected filtrates were concentrated using a rotary evaporator, producing concentrated methanol tannin extract (TME). This extract was dried in an oven overnight at 50 °C to produce a dark brown solid. The solid was stored in the refrigerator before analysis (Abdulmajid et al., 2019). The methanol tannin extract obtained was characterized using FTIR.

Preparation of iron plates

Previously in gravimetric measurements (weight loss), the surface of the iron plates (size 7.5 cm x 4 cm x 0.1 cm) was washed with a running tap of water and then polished using SiC sandpaper (grade 100, 150, 180, 240, 400 and 1200), rinsed with distilled water, dried on clean tissue paper, soaked in benzene for 5 s, dried and then soaked in acetone for 5 seconds, and dried on clean tissue paper. Finally, the clean iron plates were stored in a desiccator and weighed before use. At the end of the test, the iron plate was washed carefully with acetone and benzene, dried, and then weighed (Shivakumar et al., 2012).

Preparation of Test Solutions

a. Preparation of test solutions without inhibitors

This research uses 2 test solutions: (1) acidic solution using H_2SO_4 and (2) alkaline solution using NaOH. Each solution was made at a concentration of 0.5 M with a volume of 25 mL for each immersion of the iron plate.

b. Preparation of test solutions with inhibitors

Tannin extraction involved sonication of 0.2 grams in 0.5 mL of methanol for a brief period to achieve dissolution. Subsequently, the dissolved extract underwent dilution with a pre-prepared acid test solution until reaching a volume of 100 mL, forming the initial stock solution. This stock solution was further diluted with an acid test solution to yield concentrations of 100, 500, and 1000 ppm, serving as the test solutions. The process was replicated to prepare test solutions in the presence of inhibitors under alkaline conditions (Abdulmajid et al., 2019).

Determination of Corrosion Rate and Inhibition Efficiency

Corrosion rates were determined by taking ten pieces of iron plate; each will be given a different treatment. Two iron plates were tested as blanks by placing them in acidic (0.5 M H_2SO_4) and alkaline (0.5 M NaOH) test solutions without the addition of inhibitors (SCG methanol tannin extract). Every two iron plates were tested by adding inhibitors of various concentrations (100 ppm, 500 ppm, and 1000 ppm) to the acid and base test solutions. Then, two other iron plates were tested by adding 1000 ppm commercial tannin to the acid and base test solutions as a comparison.

The immersion time for the iron plates was five days, and ten iron plates were removed from the test solution. After that, the iron plate was sanded again with sandpaper, and the previous work method was repeated to prepare the iron plate. Then, the iron plate was weighed and measured. After cleaning, the iron plate is tested again using the same method (Yakubu, 2019).

The corrosion rate was calculated by the equation (1).

$$\text{Corrosion Rate (mmpy)} = \frac{k\,W}{A\,T\,D} \qquad (1)$$

Where:
k = constant (8.76 x 10^4)
A = Surface area (cm^2)
D = Density ($gram/cm^3$)
T = Time (hours)
W Weight loss (grams) (Sirajunnisa et al., 2014).

In calculating corrosion rate, the units commonly used are mm/year (international standard) or mill/year (mpy, British standard). A material's resistance level to corrosion generally has a corrosion rate value between 1-200 mpy.

Table 1. classified material resistance levels based on their corrosion rate (Afandi *et al*., 2015).

Table 1. Level of corrosion resistance based on corrosion rate

Relative corrosion resistance	Matrix equivalent approximation				
	mpy	mm/ year	μm/ yr	nm/ yr	pm/ sec
Excellence	<1	<0.02	<25	<2	<1
Very good	1-5	0.02-0.1	25-100	2-10	1-5
Good	5-20	0.1-0.5	100-500	10-50	5-20
Average	20-50	0.5-1	500-1000	50-100	20-50
Poor	50-200	1-5	1000-5000	150-500	50-200
Not acceptable	200+	5+	5000+	500+	200+

Inhibition efficiency was calculated using the equation (2):1% = $\frac{\omega 1}{\omega 0}$ 1 (2)

Where, ω 0 and ω 1 are the weight loss values in the absence and the presence of tannin extracts of SCG (inhibitor) (Ezeh et al., 2023). The treatment was performed for five days with two repetitions and monitored using a digital USB microscope to analyze the iron plate's surface morphology (physical barriers to corrosion) (Yetri & Handani, 2016).

RESULT AND DISCUSSION

Extraction of tannin from SCG using the maceration method using methanol solvent produced 5.1554 grams of methanol tannin extract, which was dark brown and solid. The results from the extraction of methanol tannin extract from coffee grounds can be seen in Table 2.

Table 2 shows that the yield obtained from SCG tannin was 10.30%. Several factors, such as sample size, extraction method, extraction temperature, extraction time, solvent concentration, and type of solvent used, can influence the yield of an extract from a material. The yield category was good if the value was more than 10% (Rujiyanti et al., 2011).

The methanol tannin extract of SCG was characterized using FTIR, which aimed to identify the functional groups in the sample. Identification was conducted based on the observed chemical bond vibrations in Figure 1.

Figure 1 shows the analysis of tannin compounds at wave numbers in the 4000-400 cm-1 range. Based on the FTIR results of SCG tannin isolates, it appeared that there was an absorption at a wave number of 3428.62 cm^{-1}, and commercial tannins show a wave number of 3401.61 cm-1 with solid intensity and a wide band shape, which showed that in SCG tannins extract and commercial tannin isolates there were groups O-H. The absorption of the O-H group in the research of Puspita Sari et al. (2015) was explained as being tied to aliphatic and aromatic groups due to intramolecular hydrogen bond vibrations. This was reinforced by the presence of weak and broad absorption in the wave number area of 1373.38 cm^{-1} in the SCG tannin isolate and 1344.44 cm^{-1} in commercial tannin, indicating the presence of the C-O-H group. The presence of the O-H group was also strengthened by weak and broad absorption at a wave number of 936.48 cm^{-1} in SCG tannin isolate and 959.63 cm^{-1} in commercial tannin.

The wave numbers in SCG and commercial tannin isolates were 2859.59 cm^{-1} and 2729.39 cm^{-1}, respectively, indicating the presence of aliphatic C-H groups. The presence of aliphatic C-H groups was strengthened by the presence of C-H bending with weak intensity at wave numbers 690.55 cm^{-1}, 598.92 cm^{-1}, 552.63 cm^{-1} in coffee grounds tannin isolates and 690.55 cm^{-1}, 647, 15 cm^{-1} and 554.56 cm^{-1} in commercial tannin.

Absorption bands at wave numbers 1715.76 cm^{-1} and 1659.82 cm^{-1} in SCG tannin isolate, and moderate intensity absorption bands at wave numbers 1711.90 cm^{-1} and 1613.52 cm^{-1} in commercial tannin showed the presence of a C=O ester group. The presence of an ester group strengthens the suspicion of hydrolyzed tannin. Wave numbers of 1205.56 cm^{-1} in the SCG tannin and 1207.49 cm^{-1} in commercial tannin indicated the presence of the C-O-C ether group.

The suspicion that tannin compounds were isolated from SCG waste was also strengthened by the presence of absorption at wave numbers 1555.66 cm^{-1}, 1525.76 cm^{-1} (SCG tannin isolate), and 1534.44 cm-1 (commercial tannin), which indicated the presence of C =C aromatic. The infrared spectrum above showed that SCG and commercial tannin isolates have the same characteristic functional groups, such as –O-H, aliphatic C-H, C=O ester, aromatic C=C, and C-O-H and C-O-C ether. These peaks were specific peaks of tannin compounds, mainly hydrolyzed tannin, thus strengthening the suspicion that SCG tannin isolates were proven to contain tannin compounds (Puspita Sari et al., 2015). Table 3 shows a comparison of tannin wave numbers from SCG with several related studies.

Data on corrosion rate and inhibition efficiency from adding SCG tannin methanol extract to iron plates in both acidic and alkaline media can be seen in Tables 4 and 5, respectively.

Table 4 showed that by increasing the concentration of SCG tannin methanol extract as a corrosion inhibitor on iron plates in the acid test solution, there was a decrease in the corrosion rate and an increase in inhibition efficiency. This matter was related to the number of molecules adsorbed because increasing the concentration of the extract means the number of molecules adsorbed on the surface of the iron plate also increases so that the protective layer (film) can form more perfectly (Sirajunnisa et al. 2014). The maximum concentration is 1000 ppm with an average corrosion rate of 2.2520 mmpy and an inhibition efficiency of 29.23%. Based on the table of corrosion resistance levels based on corrosion rate (Table 1), it can be concluded that the acid test solution has poor corrosion resistance. The reason was that the more acidic a solution was, the more ions were produced and the more electrolytic the solution would be. Hence, the corrosion rate was a more significant and easier way to corrosion (Pandia et al., 2021).

Table 2. SCG methanol tannin extraction

Sample	SCG weight (g)	Extract weight (g)	Yield (%)
Extract methanol tannins of SCG	50,04	5,1554	10,30

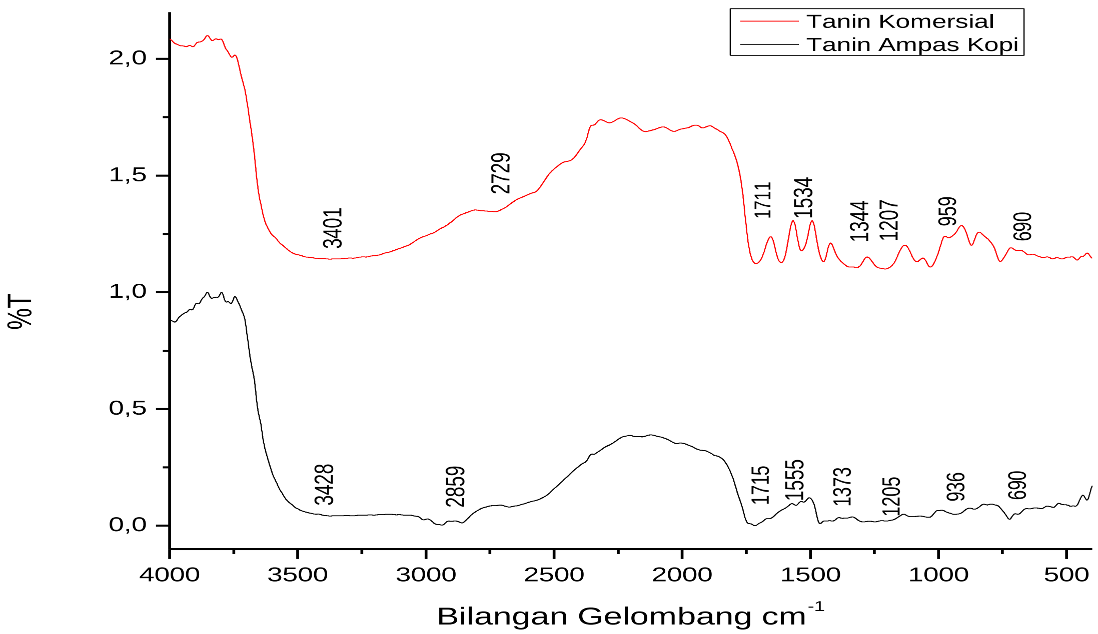

Figure 1. FTIR spectrum of SCG methanol tannin extract and commercial tannin

Table 3. The comparison of SCG methanol tannin wavenumber with several related studies

Functional groups	SCG Methanol tannin (cm^{-1})	Commercial tannin (cm^{-1})	Trembesi leaf tannin (Puspita Sari *et al*, 2015) (cm^{-1})	Tamarind shell tannin (Abdulmajid *et al*, 2019) (cm^{-1})
O-H	3428,62	3401,61	3556,74	3411
C-O-H	1373,38	1344,44	1363,67	1378
C-H aliphatic	2859,59	2729,39	2889,37	2861
C=O ester	1715,76; 1659,82	1711,90; 1613,52	1743,65	-
C=C aromatic	1555,66; 1525,76	1534,44	1516,05	1522
C-O-C ether	1205,56; 1109,12	1207,49	1215,15	1261
O-H *bending*	936,48	959,63	966,34	-
C-H *bending*	690,55; 598,92; 552,63	690,55; 647,15; 554,56	574,79; 507,28	-

Table 4. Corrosion rate and inhibition efficiency of iron plate in acid test solution (0.5 M H_2SO_4)

Inhibitor Concentration (ppm (ppm)	Corrosion Rate (mm/y)			Inhibition Efficiency (%) rata-rata
	5 Hari (1)	5 Hari (2)	Rata-rata	
0	2,9181	2.9545	2.9363	0
100	2.7020	2.8003	2.7511	6.56
500	2.5918	2.5327	2.5622	14,61
1000	2.3830	2.1211	2.2520	29.23
1000 comercial	1.9744	2.0113	1.9928	30.66

Table 5. Corrosion rate and inhibition efficiency of iron plate in alkaline test solution (0.5 M NaOH)

Inhibitor Concentration (ppm (ppm)	Corrosion Rate (mm/y)			Inhibition Efficiency (%) rata-rata
	5 Hari (1)	5 Hari (2)	Rata-rata	
0	0.1182	0.1000	0.1091	0
100	0.0883	0.0923	0.0903	8.26
500	0.0704	0.0694	0.0699	25.46
1000	0.0604	0.0579	0.0592	48.45
1000 comercial	0.0546	0.0552	0.0549	50.93

In the alkaline test solution, the maximum concentration of SCG methanol tannin extract was also 1000 ppm with an average corrosion rate of 0.0592 mmpy and the highest inhibition efficiency of 50.93% as seen in table 5. From this data, based on the table of corrosion resistance levels Based on the corrosion rate (Table 1), it can be concluded that the alkaline test solution has excellent corrosion resistance. This was because the weaker the acidity, the fewer solution ions produced, so the ability to conduct electrons will decrease and cause the corrosion rate to decrease (Pandia et al., 2021).

Iron plate surface testing was done with a USB digital microscope at 1000x magnification.

After immersion in acidic and alkaline test solutions in the presence and absence of inhibitors, iron plate specimens were taken, then dried and stored in a desiccator. Plate specimens with optimal and without inhibitor concentrations were reviewed for their surface morphology as shown in Figure 2.

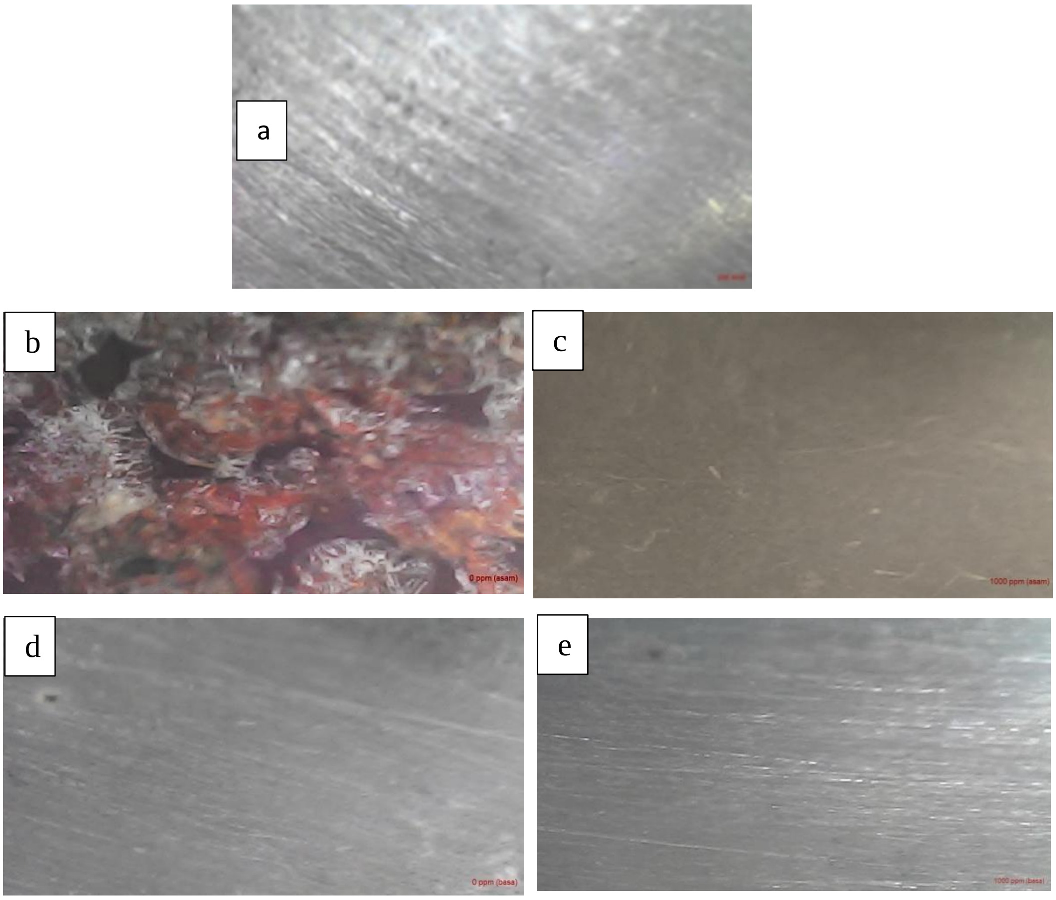

Figure 2. Iron plate surface morphology; a) initial plate, b) acid blank, c) 1000 ppm methanol tannin extract in acidic solution (0.5 M H_2SO_4), d) alkaline blank, e) 1000 ppm methanol tannin extract in alkaline solution (0.5 M NaOH)

Figure 2a shows the surface morphology of the initial iron plate before testing. Figure 2b, without the addition of a corrosion inhibitor, described that the surface morphology of the iron plate experienced heavy corrosion compared to Figure 2c, which added a corrosion inhibitor with a concentration of 1000 ppm in the acid test solution of 0.5 M H_2SO_4. Furthermore, the results of the analysis proved that iron plates were less susceptible to corrosion in the presence of SCG methanol tannin extract inhibitors. These were related to the adsorption of SCG methanol tannin extract inhibitor molecules on the surface of the iron plate, forming a protective layer so that corrosion of the plate was inhibited (Abdulmajid et al., 2019).

Figure 2d and Figure 2e did not show a significant difference in surface morphology. As the picture describes, both plates have no spots and look the same as before testing. This proved that the corrosion rate in the alkaline test solution of 0.5 M NaOH was minimal. The small value of the corrosion rate in alkaline due to the weak acidity in the solution and fewer ions produced so that the ability to conduct electrons decreases and causes the corrosion rate to also decrease (Pandia et al., 2021).

CONCLUSIONS

Based on the research that has been carried out, it can be concluded that a methanol tannin extract from coffee grounds was proven to be successful in influencing the rate of corrosion on iron plates with the corrosion inhibition efficiency of iron plates in acid and base solutions at maximum concentrations (1000 ppm) respectively, namely 29.23% and 50.93% with soaking time for five days (120 hours). The average value of the corrosion rate on iron plates with the addition of SCG methanol tannin extract inhibitor at the maximum concentration in acidic solutions was 2.2520 mmpy, which classified as flawed criteria for resistance corrosion (according to Table 1) in alkaline solutions, was 0.0592 mmpy, which classified as very good criteria for resistance corrosion (according to Table 1).

ACKNOWLEDGMENTS

The authors were highly thankful to Ir. Susilawati, M.Si, for providing an iron plate to support this research, and Dr. Muliadi Ramli, head of the inorganic. laboratory, Department of Chemistry, USK, Banda Aceh, for giving necessary research facilities in the laboratory.

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