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Effect of Kersen Leaf Extract Inhibitor Concentration and $NaNO_2$ Blending for Corrosion Protection of ASTM A53 Steel in Artificial Brine Water Media

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Abstract*—Kersen leaves contain tannin compounds that can be utilized as corrosion inhibitors against corrosion rates. Corrosion inhibitor refers to a chemical compound that, when introduced to an environment, can decrease the speed of corrosion that takes place in that environment against a metal present within it. This research aims to assess the impact of kersen leaf extract inhibitor concentration and inhibitor mixing (kersen leaf extract and NaNO2) on diminishing the corrosion rate of ASTM A53 steel in artificial brine water media. This study was conducted using the weight loss method which refers to the ASTM G31-72 standard. Kersen leaves were extracted by maceration with 96% ethanol solvent for 5 days. Inhibitors were added with various concentrations (0; 200; 400; 600; 800; 1000; 1200; 1400; 1600; 1800; and 2000) ppm. The steel plate was immersed for 7 days. The analysis carried out is the analysis of corrosion rate, inhibition efficiency, macro photos, and SEM. The findings indicated that the concentration with the minimum corrosion rate of kersen leaf extract inhibitor was achieved at a concentration of 1000 ppm, resulting in a corrosion rate of 0. 7469 mpy and an inhibition efficiency of 84. 9796%. Inhibitor blending is a concentration of 1400 ppm with a corrosion rate of 0.656 mpy and an inhibition efficiency of 84.9796%. SEM characterization showed that the specimens experienced stress corrosion cracking and pitting corrosion.*

***Keywords*—** *artificial brine water, ASTM A53 steel, blending, kersen leaf extract, corrosion inhibitor*

I. INTRODUCTION

Some natural extracts contain organic compounds that can reduce metal corrosion rates such as tannins, alkaloids, pigments, saponins, carbohydrates, and amino acids (Irianty & Khairat, 2013). One of them is kersen leaf extract. Through phytochemical tests conducted by Annisa & Najib (2022), kersen leaf extract contains phenol, flavonoid, and tannin secondary metabolite compounds. The total phenolic content of kersen leaf extract amounted to 22,389 mg of extract. Total flavonoid levels amounted to 13,375 mg of extract and tannin levels amounted to 13,715 mg of extract. Tannins are one of the organic compounds that have the ability to stop metal corrosion. Due to its properties that can form complexes with metals, tannins are also environmentally friendly organic compounds. Tannins have a very large structure for form macromolecules and contain many hydroxy groups (-OH), which is the basis that tannins have potential as corrosion inhibitors (Rochmat et al., 2019).

Research conducted by Hidayat et al., (2023) made organic inhibitors from kersen leaf extract and then applied to ASTM A36 steel in seawater media. The extraction process was carried out by maceration extraction using 96% ethanol solvent. Steel was immersed for 14 days with changes in inhibitor concentrations of 0 ppm, 100 ppm, 200 ppm, 300 ppm, 400 ppm, and 500 ppm. Analysis of corrosion rate using weight loss method. The best results were obtained at a concentration of 500 ppm with a corrosion rate of 0.77 mpy and an efficiency of 38.83%.

Research conducted by Wardani et al., (2023) applied ASTM A53 steel using inhibitors from papaya leaf extract and papaya gum with seawater media. By varying the concentration of papaya leaf inhibitor and papaya gum of 0 ppm, 200 ppm, 400 ppm, 600 ppm, and 800 ppm. Analysis of corrosion rate using weight loss method. The best results of papaya leaf extract and papaya gum were obtained at a concentration of 800 ppm with a corrosion rate of 0.5047 mm/year and 0.4509 mm/year, respectively. The inhibitor efficiency of papaya leaf is 11.64% and papaya gum is 20.82%.

Based on these studies, this study conducted corrosion testing using kersen leaf extract which varied its concentration on ASTM A53 steel plate with artificial brine water media. In addition, variations in the concentration of alternative inhibitors were also carried out, namely blending between organic inhibitors (kersen leaf extract inhibitor) and inorganic inhibitors, namely NaNO2. Research by Juanda et al. (2022) proved that the addition of NaNO2 inhibitor can reduce the corrosion rate.

The aim of this research was to assess the impact of differing inhibitor concentrations of kersen leaf extract and NaNO2 mixture for preventing corrosion rates of ASTM A53 steel in synthetic brine water environment.

The benefit of this research is to utilize kersen leaves into a corrosion inhibitor that can reduce corrosion rates that are environmentally friendly and have more use value.

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II. METHODOLOGY

This research started from September to November 2024 at the Research Laboratory of the Chemical Engineering Department of Samarinda State Polytechnic. Kersen leaves as raw materials were obtained from several houses in the Samarinda city area. The location of steel plate preparation and inhibitor manufacturing was carried out in the Instrumentation Laboratory of the Chemical Engineering Department of Samarinda State Polytechnic.

A. Fixed Variable

1. Steel Coupon Type : ASTM A53
2. Soaking Media: Artificial Brine Water
3. Soaking Volume: 1000 mL
4. Maceration Time : 5 days
5. Soaking Time: 7 days

B. Variable Change

1. Kersen leaf inhibitor concentration: (0 ; 200 ; 400 ; 600 ; 800 ; 1000 ; 1200 ; 1400 ; 1600 ; 1800; and 2000) ppm
2. Concentration of $NaNO_2$ blending inhibitor and kersen leaf inhibitor: (0 ; 200 ; 400 ; 600 ; 800 ; 1000 ; 1200 ; 1400 ;1600 ; 1800 ; and 2000) ppm in a 1:1 ratio

C. Response Variable

1. Corrosion Rate (weight loss)
2. Inhibitor Efficiency
3. Macro Photo Analysis
4. SEM Analysis

The research procedure is divided into 3, namely: Raw material preparation

1. Take 2000 grams of kersen leaves and dry them in the sun.
2. Smoothing leaves Kersen using a blender and chopper.
3. Soaking the fine kersen leaf powder with 96% ethanol as much as 4 liters.
4. Allow the solution to stand for 5 days.
5. Filter the results of soaking using a sieve and take the resulting filtrate.
6. Concentrate the filtrate using a vaccum rotary evaporator at 40°C to obtain solvent-free concentrated kersen leaf extract.
7. Store the extract product into the prepared sample container.
8. Take 500 mg of extract for qualitative tannin test.
9. Dilute the extract with distilled water until almost colorless.
10. Take 2 mL and then add 2 drops of FeCl3 10%. The solution will change color to greenish indicating the extract contains tannins.

Procedure for making Artificial Brine Water solution

1. Put 20 grams of NaCl and 0.2 grams of $NaHCO_3$ into a 1000 mL volumetric flask.
2. Dissolve all ingredients by adding distilled water into a 1000 ml volumetric flask until the limit mark then shaken.

ASTM A53 Steel Coupon Preparation

1. Prepare the steel plate to be used.
2. Cleaning the plate from dirt attached to the plate using concentrated HCl.
3. Sanding the plate using sandpaper with sizes 600, 800, and 1000 grit until the surface of the plate is smooth.
4. Wash the plate thoroughly using soap
5. Rinse the plate using running water and then dry it.
6. Numbering each plate both blank and test sample.
7. Weigh the initial mass of each plate using a digital balance ($_{W0}$).
8. Measure the length, width, thickness, and diameter of the plate using a digital caliper.

The Analysis Test Procedure is divided into 2, namely:

A. Weight loss test procedure with extract inhibitor kersen leaves

1. Prepare the plate that has been prepared.
2. Make the bath test solution as follows:
   - Adding kersen leaf extract as much as (0.2 gram, 0.4 gram, 0.6 gram, 0.8 gram, 1 gram, 1.2 grams, 1.4 grams, 1.6 grams 1.8 grams, and 2 grams) into a 1000 mL volumetric flask.
   - Mixing solution with artificial brine water until homogeneous
   - Transfer into the sample container that has been provided
3. Hook the thread that has been tied to the steel plate in the center of the stick.
4. Number the containers to facilitate observation.
5. Immerse the plate tied with thread and stick into a container that contains artificial brine water that has been added with kersen leaf extract inhibitor.
6. Insert a stick into the top side of the perforated container to prevent it from moving.
7. Allow the plate to soak for 7 days.
8. Analyze the corrosion rate using the macro photo method as follows:
   - Remove the test plate from the test environment every 2 days and take pictures of the test plate.
   - Submerging the captured plate back into the test environment
9. Remove the plate from the bath after the soaking time has expired.
10. Washing the plate with running water, drying, and weighing the final mass of the plate using a digital balance (W1).
11. Calculate the mass of the missing plate (W).
12. Calculate the corrosion rate that occurs.

B. Weight loss test procedure with inhibitor blending of kersen leaf extract with $NaNO_2$

1. Prepare the plate that has been prepared.
2. Make the bath test solution as follows:

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- Adding inhibitor blending of kersen leaf extract and NaNO2 in a ratio of 1:1 as follows:
  - Kersen leaf extract as much as (0.1 gram, 0.2 gram, 0.3 gram, 0.4 gram, 0.5 gram, 0.6 gram, 0.7 gram, 0.8 gram 0.9 gram, and 1 gram) into a 1000 mL volumetric flask.
  - Adding NaNO2 as much as (0.1 gram, 0.2 gram, 0.3 gram, 0.4 gram, 0.5 gram, 0.6 gram, 0.7 gram, 0.8 gram 0.9 gram, and 1 gram) into a 1000 mL volumetric flask.
- Mixing solution with artificial brine water until homogeneous
- Transfer into the sample container that has been provided

3. Hook the thread that has been tied to the steel plate in the center of the stick.
4. Number the containers to facilitate observation.
5. Soaking the bonded plate with thread and stick into a container containing artificial brine water that has been added with kersen leaf extract inhibitor.
6. Insert a stick into the top side of the perforated container to prevent it from moving.
7. Allow the plate to soak for 7 days.
8. Analyze the corrosion rate using the macro photo method as follows:
9. Remove the test plate from the test environment every 2 days and take pictures of the test plate.
10. Submerging the captured plate back into the test environment
11. Remove the plate from the bath after the soaking time has expired.
12. Washing the plate under running water, drying, and weighing the final mass of the plate using a digital balance (W1).
13. Calculate the mass of the missing plate (W).
14. Calculate the corrosion rate that occurs.

III. RESEARCH RESULTS

TABLE 1. Corrosion rate with Kersen Leaf Extract Inhibitor

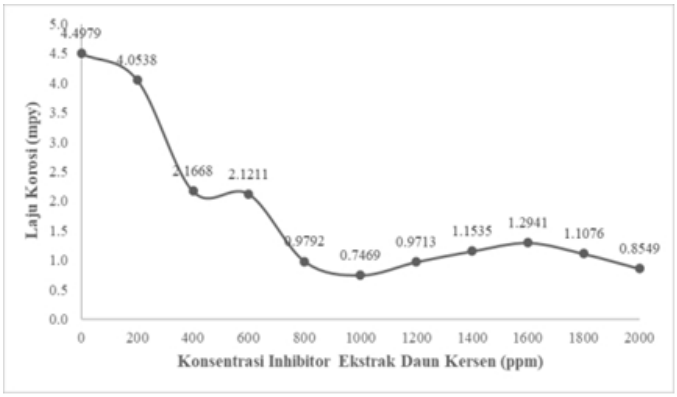
Concentration (ppm)	Initial Mass (mg)	Final Mass (mg)	Weigh t Loss (mg)	Corro sion Rate (mpy)	Inhibition Efficiency (%)
0	18767,4	18730,7	36,7	4,4979	0
200	18078,3	18046,3	32,0	4,0538	9,8735
400	18712,5	18694,2	18,3	2,1668	51.8264
600	19398,0	19380,1	17,9	2,1211	52,8423
800	17382,5	17374,7	7,8	0,9792	78,2298
1000	19142,3	19136,1	6,2	0,7469	83,3944
1200	19130,6	19122,5	8,1	0,9713	78,3921
1400	20210,9	20200,3	10,6	1,1535	74,3546
1600	20176,3	20165,2	11,1	1,2941	71,2287
1800	19012,7	19003,4	9,3	1,1076	75,3751
2000	18642,7	18635,6	7,1	0,8549	80,9933

IV. DISCUSSION

Effect of Kersen Leaf Extract Inhibitor Concentration and Inhibitor Blending on Corrosion Rate

TABLE 2. Corrosion Rate with Inhibitor Blending between Kersen Leaf Extract and $NaNO_2$

Concentration (ppm)	Mass Initia l (mg)	Mass End (mg)	*Weight Loss* (mg)	The pace Corros ion (mpy)	Efficiency Inhibiti on (%)
0	18767,4	18730,7	36,7	4,4979	0
200	17491,8	17457,6	34,2	4,3124	4,1241
400	18562,6	18539,3	23,3	2,8576	36,4681
600	19217,5	19202,3	15,2	1,8227	59,4766
800	20350,9	20338,6	12,3	1,3295	70,4417
1000	19408,2	19400,8	7,4	0,8994	80,0040
1200	20981,6	20971,7	9,9	1,1198	75,1039
1400	18462,8	18457,3	5,5	0,6756	84,9796
1600	17380,0	17371,9	8,1	1,0137	77,4628
1800	17262,4	17252,5	9,9	1,2547	72,1047
2000	20572,8	20564,8	8,0	0,9061	79,8550



Analysis of Corrosion Rate of Kersen Leaf Extract Inhibitor

Figure 1. shows that with the addition of kersen leaf extract inhibitor concentration, the corrosion rate decreases. Based on the results obtained, the corrosion rate without the addition of inhibitors in Artificial Brine Water media of 4.4979 mpy can be seen in table 4.1. After the addition of the inhibitor, the lowest corrosion rate obtained in this study was at a concentration of 1000 ppm with a corrosion rate of 0.7469 with an efficiency of 83.3944%, this is because the hydrophobic layer formed is perfect which is characterized by covering the entire surface of the steel plate by the inhibitor. There are several points that tend to increase the corrosion rate, namely at concentrations of 1200 ppm, 1400 ppm, 1600 ppm, and 1800 ppm. This is due to the slight peeling of the hydrophobic layer on the test plate, so that the test plate is in direct contact with the corrosive media. Then at a concentration of 2000 ppm the corrosion rate decreased again to 0.8549 mpy with an efficiency of 80.9933%, this is because the inhibitor works again to protect the surface that has undergone peeling.

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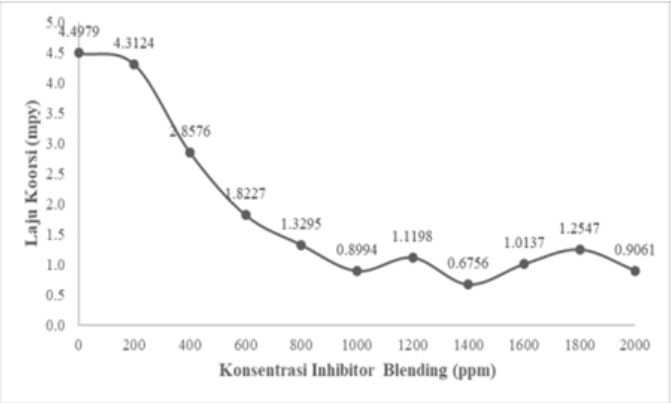
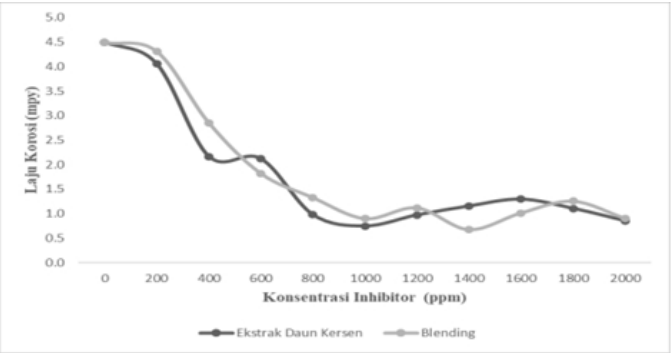


Corrosion Rate Analysis of Blended Inhibitors

Based on Figure 2. shows that the higher the concentration of inhibitor, the lower the corrosion rate. This can be seen at a concentration of 200 ppm to 1000 ppm the corrosion rate decreases but at a concentration of 1200 ppm the corrosion rate increases and decreases again at a concentration of 1400 ppm. Then increased at concentrations of 1600 ppm and 1800 ppm and decreased again at a concentration of 2000 ppm. The increase in corrosion rate is due to the weak adsorption binding power, the surface of the plate in direct contact with the corrosive media that has previously been coated by the inhibitor is partially peeled off. This can be seen from the sediment in the media solution that causes the corrosion rate to increase.

The peeled surface loses its protection to prevent corrosion. Then as the corrosion rate decreases again, it shows that inhibitor is working again to protect the peeled surface.

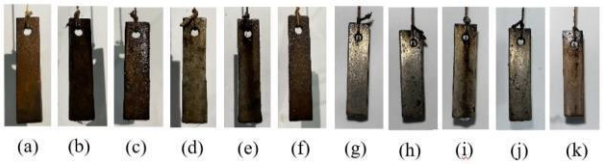


Comparison of Corrosion Rate of Kersen Leaf Extract Inhibitor and Blending Inhibitor

Figure 3. shows the results of the comparison of corrosion rates using kersen leaf extract inhibitors and blending inhibitors between kersen leaf extract and sodium nitrite. From the results of the research that has been done, it is found that the corrosion rate of blending inhibitors can reduce the corrosion rate better than kersen leaf extract. Due to each inhibitor, where kersen leaf extract forms a hydrophobic layer and contains tannin compounds and the addition of sodium nitrite can reduce the corrosion rate because sodium nitrite has a function as an oxygen trap, which is the main factor causing corrosion in an immersion environment.

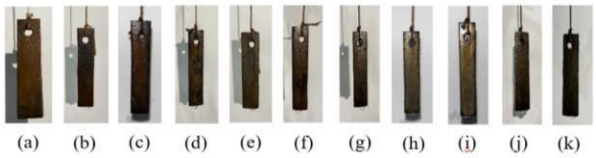
V. Macro Photo Analysis

Macro photo analysis was carried out to determine the growth of corrosion on the sample and to compare the corrosion growth of samples without inhibitors and using different inhibitors so that the characteristics of corrosion that occurred in steel samples were known before immersion. Macro photo analysis in this study was conducted every 2 days.



Macro Photographs of Kersen Leaf Extract Inhibitor on ASTM A53 Steel (a) 0 ppm; (b) 200 ppm; (c) 400 ppm; (d) 600 ppm; I 800 ppm; (f) 1000 ppm; (g) 1200 ppm (h) 1400 ppm; (i) 1600 ppm; (j) 1800 ppm; (k) 2000 ppm.

Figure 4. shows that corrosion is only visible in the blank, while in the addition of kersen leaf extract inhibitor corrosion appears but only a small part of it. This is due to the kersen leaf extract forming a black hydrophobic layer. If through eye sight, corrosion on steel samples is not visible because the layer is solid black, therefore further analysis is needed to determine the point of corrosion that occurs in these samples. However, at concentrations of 1200 ppm to 2000 ppm, there is a lack of adsorption layer on the surface of the plate due to the saturation of the inhibitor layer, thus making the surface of the plate black previously coated by the inhibitor undergoes peeling.



Macro Photograph of Blending Inhibitor on ASTM A53 Steel (a) 0 ppm; (b) 200 ppm; (c) 400 ppm; (d) 600 ppm; I 800 ppm; (f) 1000 ppm; (g) 1200 ppm (h) 1400 ppm; (i) 1600 ppm; (j) 1800 ppm; (k) 2000 ppm

Figure 5. shows that the inhibitor works well characterized by the adsorption layer attached to the surface of the test plate. The inhibitor used is a blending between kersen leaf extract and sodium nitrite, where kersen leaf extract forms a black hydrophobic layer to inhibit corrosion from entering directly on the entire steel surface, while sodium nitrite which is responsible for reducing oxygen by forming a passive layer that cannot be seen by eye sight can inhibit corrosion. At concentrations of 200 ppm to 1400 ppm, the surface of the plate looks formed and covered by the inhibitor, at concentrations of 1600 ppm and 1800 ppm there is a lack of adsorption layer on the surface of the plate. This is due to the peeling of the hydrophobic layer on the test plate. At a concentration of 2000 ppm the surface of the plate is again covered by the inhibitor, this is because the inhibitor again works to protect the surface that has experienced peeling.

SEM (Scanning Electron Microscopy) Analysis

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SEM testing is used to see the shape of corrosion that occurs after testing on the surface of ASTM A53 steel. The magnifications used are 210×, 500× and 5000×. The results of SEM testing can be seen in the picture below:

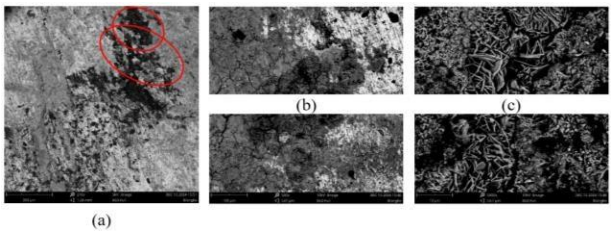

Figure 6. SEM Test Results without Inhibitor (a) 210× Magnification; (b) 500× Magnification; (c) 5000× Magnification

Figure 6. shows the SEM results of the steel blank ASTM A53 magnification of 210×, 500× and 5000× exposed to corrosion with artificial brine water media. At 210× magnification, it is clearly visible the presence of clumps (clusters) of varying sizes that are scattered. After being corroded with artificial brine water media there is a change in appearance, the presence of a dark colored image shows the area affected by corrosion. At a magnification of 500× the clusters look clearer and there are cracks and holes formed. Cracks and holes are the main factors causing corrosion, as they are the entry point for oxygen. At 5000× magnification, the colored areas The dark areas are more evenly distributed and the holes formed on the sample are almost evenly distributed on its surface and the size of the holes is already in the large category. Changes in the appearance of dark colored areas indicate areas affected by corrosion, often referred to as pitting corrosion. The SEM results also identified that the steel had been subjected to stress corrosion cracking. The characteristic of stress corrosion cracking is the presence of cracks that occur on the surface of the steel.

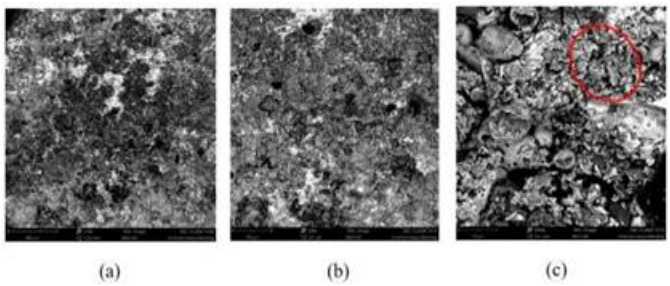

Figure 7. SEM Test Results of Kersen Leaf Extract Inhibitor (a) 210× magnification; (b) 500× magnification; (c) 5000× magnification.

Figure 7. with a magnification of 210× shows the general shape of the material surface. Black dots indicate irregularities on the surface of the material. At a magnification of 500× the surface of the material looks more detailed with this magnification the surface structure can be seen cracks (crack) and holes (hole). At a magnification of 5000×, cracks and some holes in the steel can be seen more clearly. At 5000× magnification pitting corrosion can also be seen, a type of corrosion in which small holes similar to wells are scattered on the metal surface, marked with red circles. The impact of pitting corrosion does not occur over a large area, but only at one point and continues to form deep depressions, causing an expansion of the corroded metal area. There are cracks between particles and as a result the pores widen and are easily penetrated by corrosive atoms, if this is allowed to continue the specimen will become porous. Deterioration due to corrosion is shown with a black image that forms like a depression or hole (Ornelasari, 2015). The crack at 5000× magnification identifies that the steel has undergone corrosion with a type of tension crack. When compared to the blank, the corrosion area characterized by darkening of the surface is less. The dark surface area on the blank is more evenly distributed than the kersen leaf extract inhibitor.

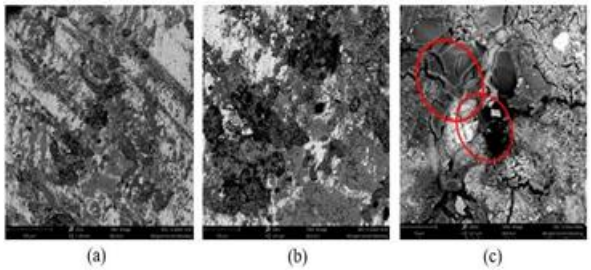

Figure 8. SEM Test Results of Inhibitor Blending (a) 210× Magnification; (b) 500× Magnification; (c) 5000× Magnification

Figure 8. shows the surface shape of the material at 210x magnification. Black lines and dots indicate irregularities on the surface of the material. The visible scratches are caused by the steel cutting process. The cutting process with burrs creates scratches that can affect the steel surface. These scratches are usually small, but they can create weak spots that accelerate corrosion. At 500x magnification, the surface is shown in more detail, and the darker surface color indicates areas affected by corrosion. At 5000x magnification, cracks and pits in the steel are more clearly visible. However, the number of holes formed is relatively less compared to the blank and when using kersen leaf extract inhibitor. The visible holes indicate that pitting (well) type corrosion has occurred on the steel, and the visible cracks indicate that stress corrosion cracking has occurred on the steel. Stress corrosion cracking is characterized by the formation of cracks on the steel surface.

VI. CONCLUSION

1. This study shows that increasing the concentration of kersen leaf extract inhibitor causes the corrosion rate to decrease and the inhibition efficiency to increase. The best concentration of kersen leaf extract inhibitor is 1000 ppm with a corrosion rate of 0.7469 mpy with an inhibition efficiency of 83.3944%.
2. Increasing the concentration of blending inhibitors (kersen leaf extract and $_{NaNO2}$) causes the corrosion rate to decrease and the inhibition efficiency to increase. The best blending inhibitor concentration is 1400 ppm with a corrosion rate of 0.6756 mpy and an inhibition efficiency of 84.9796%.

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