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EXTRACTION OF SOURSOP LEAVES (*ANNONA MURICATA* L.) AS A SOURCE OF ZINC CORROSION INHIBITOR IN SODIUM CHLORIDE MEDIUM

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Currently, corrosion inhibitors derived from organic materials are preferred in the industry because organic inhibitors are environmentally friendly and cheaper than inorganic inhibitors. This study was aimed at determining the corrosion inhibition ability of soursop leaf extract (*Annona muricata* L.) towards zinc. Soursop leaves extraction was carried out by maceration method using ethanol as solvent. In this study, the variations in the ratio of the soursop leaves powder weight to solvent volume were 1:4 g/ml, 1:6 g/ml, 1:8 g/ml, and 1:10 g/ml, and the immersion duration was two days. The extract obtained was studied as a zinc corrosion inhibitor using a 3% sodium chloride solution as the immersion medium. The inhibitor concentration was 2%, with the immersion duration varying from 2 days to 8 days. The results obtained showed that at the ratio of the soursop leaves powder weight to solvent volume of 1:10, the lowest corrosion rate (2.28 mpy) and inhibition efficiency (61.20%) were observed after eight days of immersion.

Keywords*:* alkaloid, maceration, soursop leaf, corrosion, organic inhibitor.

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Introduction

Soursop (*Annona muricata* L.) is a large tree that thrives in Indonesia, and this plant is often studied mainly for its leaves which have many benefits. Soursop leaves contain alkaloids, terpenoids, flavonoids, coumarins, steroids, fatty acids, phlobatanins, phenolics, tannins, and saponins [1]. Soursop leaves have many benefits in various fields, for example, in the health sector. Soursop leaves contain the largest compounds, namely alkaloids with a list of phytocomponents in the form of 6-hydroxyundulatine ($C_{22}H_{28}N_2O_3$) of 22.80%, stylopine ($C_{19}H_{17}NO_4$) of 20.00%, 3-isobutyryloxy-6-hydroxytropane ($C_{12}H_{21}NO$) of 5.70%, and 13-α benzoyloxylupanine ($C_{22}H_{28}N_2O_3$) of 2.32% [2].

Alkaloids are a collection of chemical compounds that contain a nitrogen atom. The term alkaloid arises from the word «alkali», which is used as a term for natural nitrogen content [3]. One way to extract these compounds is to use extraction techniques. Extraction is a process of taking components of chemical compounds, one of which can be done by the maceration method. This method offers a process that is easy to do with simple equipment. The process of working on maceration method is by soaking natural ingredients with organic solvents in a container for a certain period of time. Researchers often do the use maceration method as a method of extraction.

Corrosion is a complex phenomenon that arises due to the interaction between water and metal surfaces, and many fields suffer losses due to corrosion, such as industry and agriculture. To overcome this problem, efforts are made to reduce the corrosion rate by using corrosion inhibitors. Corrosion inhibitors generally come from synthetic chemicals, which are hazardous chemicals, relatively expensive, and not environmentally friendly. In order not to damage the environment, inhibitors derived from extracts of natural ingredients are used, especially compounds containing N, O, P, and S atoms, and atoms having lone pairs of electrons. Natural organic inhibitors are more biodegradable and easier to obtain than synthetic organic inhibitors. Research on the use of extracts of natural ingredients as corrosion inhibitors has been widely carried out [4]. Other researchers have also used plant extracts from the *Annona* family as corrosion inhibitors, such as *Annona Squamosa* [5] and *Annona cherimola* [6]. This study was aimed at determining the effect of soursop leaf extract on the corrosion rate of zinc. Soursop leaves extraction was carried out by maceration method using ethanol as solvent. In this study, the soursop leaves used were made into a powder so that the soursop leaves particles could interact optimally in the extraction process.

Experimental

This study uses soursop leaves as inhibitor corrosion and ethanol as a solvent. The metal sample is a zinc plate of 2×1×0.16 cm and 3% sodium chloride (NaCl) as a corrosive medium. The composition of the zinc plate can be seen in Table (before immersion). The Fourier transformed infrared spectroscopy (FTIR spectroscopy) is used to identify the functional group of soursop leaves. The scanning electron microscopy and energy dispersive X-ray analysis (SEM-EDX) is used to characterize the morphology and chemical composition of zinc plate surface.

Composition of zinc plates before and after immersion

Element	Composition before immersion (%)	Composition after immersion (%)	
		without inhibitor	using soursop leaves extract
Zn	95.8	70.2	80.0
C	4.2	17.4	15.1
O	–	9.0	4.9
Pb	–	3.0	–
Nb	–	0.4	–

Making soursop leaves powder

Soursop leaves are washed to remove dirt on the leaves and then dried under the sunlight for three days. Next, soursop leaves are blended until smooth and sieved to obtain a sample size that passes the 50/70 mesh sieve tray. The soursop leaves powder obtained is stored in a container for the extraction process.

Soursop leaves powder extraction process

The container of the extraction process takes place is prepared then soursop leaves powder with a particle size of 50/70 mesh and 96% ethanol solvent is put into a container with a ratio of the weight of samples to solvent volume equal to 1:4 g/ml, 1:6 g/ml, 1:8 g/ml, and 1:10 g/ml. The extraction process by immersion was carried out for two days. After the extraction process, the sample is removed from the container and filtered using filter paper. Furthermore, the solvent in the filtrate was reduced using a rotary vacuum evaporator at a temperature of 65^{0}C for 2 hours. Finally, the extracts obtained were each put in a glass bottle to be applied as a corrosion inhibitor.

Corrosion testing procedure on zinc plate

The zinc plate with a size of 2×1×0.16 cm is smoothed with sandpaper. The zinc plate is washed and dried, then weighed and recorded as the initial mass of the zinc plate. A 3% NaCl solution is poured as much as 50 ml into a beaker glass, and a zinc plate is inserted as an immersion without inhibitor into the corrosion medium. The zinc plates are immersed for two days, four days, six days, and eight days. After immersion, the zinc plate is removed and then washed, dried, and weighed to be recorded as the final mass. In the immersion of the zinc plate using a corrosion inhibitor, the zinc plate is smeared with an inhibitor of soursop leaf extract with a concentration of 2% based on the ratio of the samples and solvents. The zinc plates are immersed for two days, four days, six days, and eight days. After the corrosion process has been running for a certain time, the corrosion product is removed from the corrosion media, then washed and dried in an oven at 110^{0}C, then weighed as the final weight [7].

Determination of corrosion rate and corrosion inhibition efficiency of zinc plate

The corrosion rate can be calculated using the following equation:

$$CR = \frac{534 \cdot W}{D \cdot A \cdot t}, \qquad (1)$$

where CR is the corrosion rate (miles/year or mpy); 534 is the coefficient; W is the difference in mass before and after immersion (mg); D is the density of the iron plate (g/cm^3); A is the surface area of the zinc plate (in^2); and t is immersion time (hours). After obtaining the corrosion rate data, the efficiency of corrosion inhibition on the zinc plate is calculated by equation 2 [8].

$$IE = \frac{CR_0 - CR_1}{CR_0} \cdot 100\%, \qquad (2)$$

where IE is the inhibition efficiency (%); CR_0 is the corrosion rate before adding the inhibitor; and CR_1 is the corrosion rate after adding the inhibitor.

Qualitative analysis of soursop leaves extract

Mayer's test

Soursop leaf extract is taken as much as 2 mg and then put into a test tube. A few drops of Mayer's reagent are dropped into a test tube. The solution forms a white or pale yellow color indicating the presence of alkaloids.

Dragendorff's test

Soursop leaf extract is taken as much as 2 ml and put into a test tube. Next, 3–5 drops of Dragendorff's reagent is poured into a test tube. If the solution changes color to orange, then the solution contains alkaloids.

Results and discussion

Alkaloids identification from soursop leaf extract

Soursop leaves extract qualitative test using the Mayer test and Dragendorff tests showed the presence of alkaloids in soursop leaves. Figure 1a shows the color change in the alkaloid test using the Mayer method, marked by the formation of a precipitate and a change in the color of the soursop leaf extract to white or pale yellow after being dropped by a few drops of Mayer's reagent. Figure 1b shows the formation of color deposits to orange or orange-red in soursop leaf extract on alkaloid testing by the Dragendorff method by adding Dragendorff's reagent.

Result of Fourier transform infrared spectroscopy analysis on soursop leaves powder

The results of the analysis of soursop leaves powder by FTIR are presented in Fig. 2.

Based on the results obtained, there is the presence of a hydroxyl functional group (O–H, H-bonded) at a wavelength of 3389.51 cm^{-1}, a functional group N–H stretch at a wavelength of 3389.51 cm^{-1}, a C–H stretch functional group and an O–H functional group at a wavelength 2922.2 cm^{-1}, a

a b

Fig. 1. Phytochemical test for alkaloid identification in soursop leaf extract: a – Mayer's test; and b – Dragendorff's test

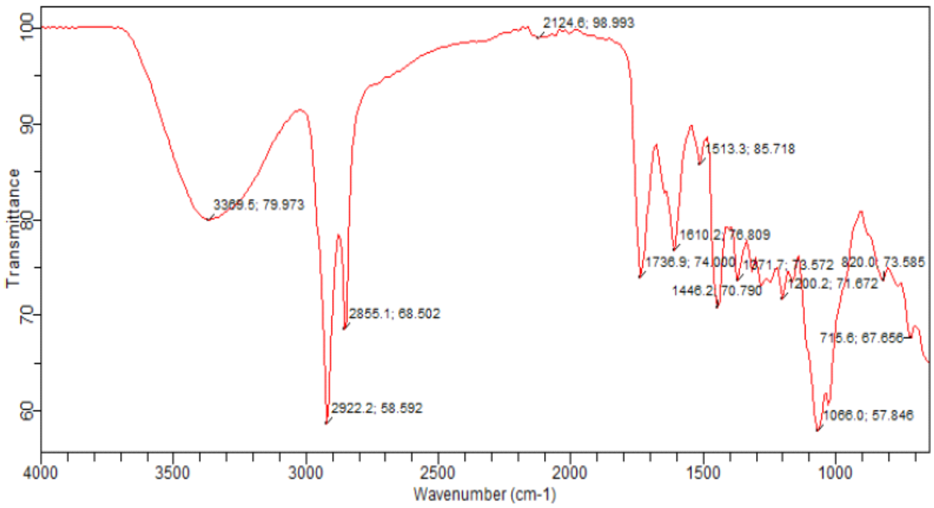


Fig. 2. Soursop leaves extract analysis by using FTIR

functional group C=C and a functional group N–H bend at a wavelength of 2855.1 cm^{-1}, a functional group C–H at a wavelength of 1736.9 cm^{-1}, functional groups C–O and C–N at a wavelength of 1200.2 cm^{-1} and a C–O functional group at a wavelength of 1066.0 cm^{-1}. The presence of a N–H functional group indicates that the soursop leaves extract contains alkaloid compounds.

The O–H and N–H functional groups are polar functional groups, whereas the C=C functional group is a heteroatom functional group. In general, the heteroatom functional group acts as an adsorption center (interacts with the metal surface) and the multiple functional groups gain electrons and gain them at the adsorption center through conjugation. The interactions between organic inhibitors (or filming inhibitors) form the surface film over the interfaces of metal and environments (or electrolytes) that isolate the metals from corrosive surrounding and can protect from corrosion [9].

Effect of ratio of amount of solvent in extraction of soursop leaves

Figure 3 shows the effect of the ratio of the amount of solvent in the extractive compounds isolated from of soursop leaves. This study used the maceration method to extract soursop leaves using ethanol as a solvent. In this study for comparison, we used the following ratios of the weights of samples to solvents: 1:4 g/mL, 1:6 g/mL, 1:8 g/mL, 1:10 g/mL, the weights of extract being 1.52 g, 2.31 g, 3.34 g, 4.17 g, respectively. The extraction results obtained by the maceration method indicate that the more solvents used, the more extracts produced. The best yield of 4.17 g is obtained at the ratio of soursop leaves and solvent as much as 1:10 g/mL with an extraction time of two days.

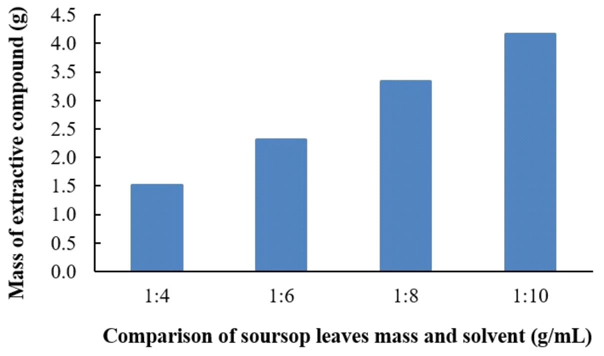


Fig. 3. The effect of the ratio of the amount of solvent in the extraction of soursop leaves

Effect of addition of soursop leaves extract inhibitor on corrosion rate of zinc

Based on the results obtained (Fig. 4), the corrosion rate decreased with the addition of the soursop leaves extract inhibitor. The determination of the corrosion rate was carried out at an eight-day immersion time as the longest immersion time. In this study, the best results were obtained at the ratio of sample weight to solvent of 1:10 g/mL on the eighth day, exhibiting the corrosion rate of 2.28 mpy. This result is in accordance with the idea that the longer the immersion time, the lower the corrosion rate. This is due to the inhibitory activity of soursop leaf extract, which reduces the corrosion rate of zinc. This process occurs due to the binding of inhibitor molecules of soursop leaf extract to the surface of zinc. Soursop leaf extract molecules suppress the reaction of metal with the medium, forming a protective layer, so that the metal surface is separated from the corrosion medium [10]. Hence, soursop leaf extract can be used to protect zinc from corrosion attacks.

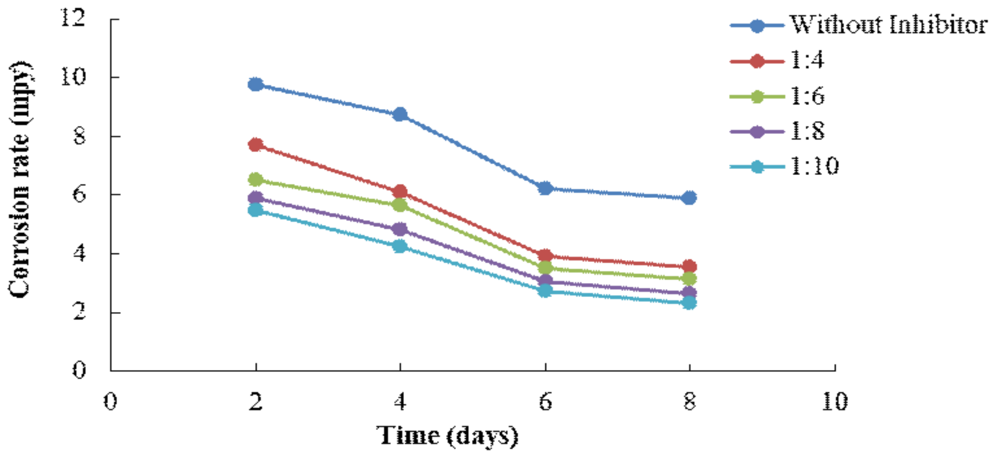


Fig. 4. Effect of addition of soursop leaf extract inhibitors on corrosion rate of zinc

Effect of addition of soursop leaves extract on efficiency of zinc corrosion inhibition in sodium chloride medium

Based on the results of the study (Fig. 5), it was found that the higher the inhibitor concentration used and the longer the immersion time, the higher the inhibition efficiency. The increase in the inhibition efficiency is due to several molecules being adsorbed on the surface of zinc and making it act as a protective film [11]. Soursop leaves extract inhibitor with a ratio of sample weight to solvent ratio of 1:10 g/mL gives the best inhibition efficiency of 61.20%. The results obtained are comparable to the work [12], which showed the efficiency of 54.50% using tea leaves as a zinc corrosion inhibitor, and the work [13], which showed the efficiency of 60.12% using petai extract as a zinc corrosion inhibitor.

Results of surface analysis and chemical composition of zinc plate samples

SEM testing was carried out to determine the surface topography of the specimen, and EDX testing was carried out to determine the chemical components contained in the sample. The SEM-EDX used has a magnification of ×1000. Figure 6 shows the results of the SEM analysis on the zinc plate surface.

Figure 6a shows the SEM image of the zinc plate before immersion. In Fig. 6a, it can be seen that the surface of the zinc plate is still smooth because the environment has not influenced the plate. Figure 6b shows the SEM image of the zinc plates after immersion without the addition of inhibitors on the sixth day. It is seen that the zinc plate surface has many large holes and scratches, and the Zn content decreased drastically to 70.2%, as shown in Table. Figure 6c shows the SEM image of the zinc plate after being immersed with the addition of soursop

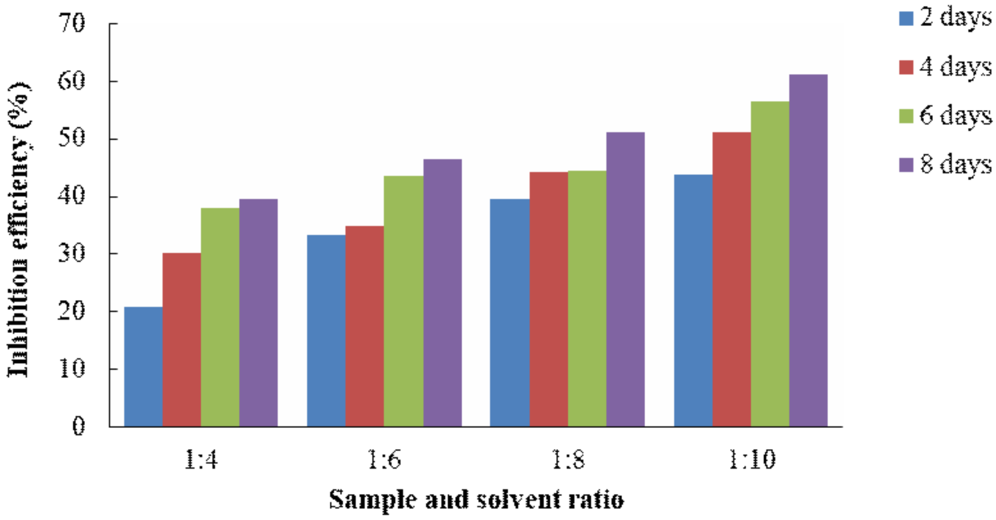
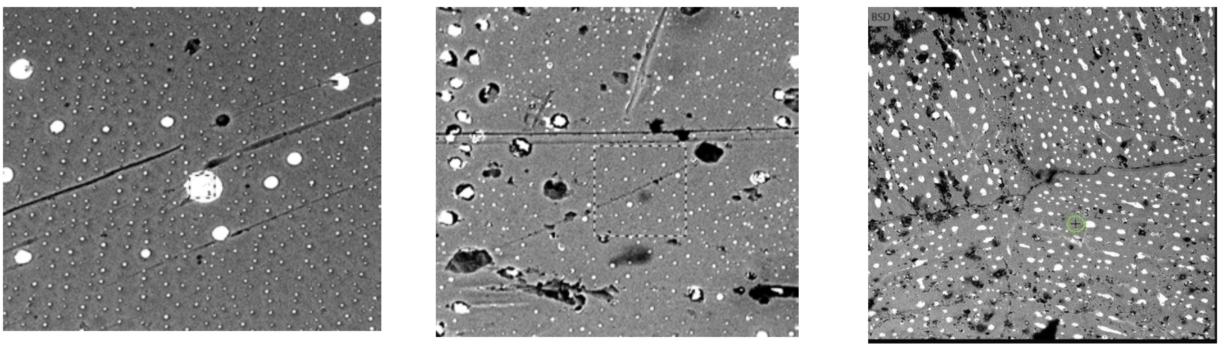


Fig. 5. Effect of addition of soursop leaves extract inhibitor on the efficiency of zinc corrosion inhibitor



a b c

Fig. 6. SEM images of the surface of zinc plate: a – before immersion; b – after immersion without inhibitor; and c – after immersion with soursop leaves extract inhibitor

leaves extract inhibitor derived from a 1:4 ratio of samples weight to solvent volume on the sixth day. In Fig. 6c, it can be observed that there are only a few scratches on the surface of the zinc plate. The selection of SEM testing on the use of inhibitors derived from a ratio of sample weight to solvent volume of 1:4 aims to predict the minimal ability of the soursop leaves extract inhibitor to protect the zinc plate as a whole from corrosion. Table shows the chemical composition of the surface of zinc plate using EDX analysis.

The zinc plate used has a purity of 95.8%. The presence of elemental carbon was increased, and there is the presence of elemental oxygen after immersion without inhibitors. This is due to the corrosion products that are formed, decreasing the content of Zn and increasing the contents of C and O. The reaction scheme that occurs is shown in Eqs. (1) and (2). The cathode half-reaction corresponds to the reduction of oxygen:

$$O_2+2H_2O+4e^-\rightarrow 4OH^-, \quad (1)$$

The anode half-reaction involves dissolving metallic zinc:

$$Zn\rightarrow Zn^{2+}+2e^-. \quad (2)$$

Both cathode and anode half-reactions imply that zinc cations (Zn^{2+}) (anode half-reaction) and hydroxide anions (OH^-) (cathode half-reaction) react to produce zinc hydroxide ($Zn(OH)_2$) according to Eq. (3) [14]:

$$Zn^{2+}+2OH^-\rightarrow Zn(OH)_2. \quad (3)$$

In the corrosive medium of sodium chloride, chloride ions (Cl^-) can migrate to the anodic sites to form zinc hydroxide chloride ($Zn_5(OH)_8Cl_2\cdot H_2O$), as shown in Eq. (4) [15].

$$5Zn(OH)_2+2Cl^-+H_2O\rightarrow Zn_5(OH)_8Cl_2\cdot H_2O+2OH^-. \quad (4)$$

Sodium ions (Na^+) in the cathodic region also react with carbonate ions to form sodium carbonate (Na_2CO_3). Therefore, some of the carbon detected on the zinc surface is due to sodium carbonate on the sample surface:

$$2Na^++CO_3^{2-}\Leftrightarrow Na_2CO_3. \quad (5)$$

In this study, soursop leaves extract as a corrosion inhibitor has a role in slowing down the corrosion rate on zinc:

$$Zn^{2+}+2Cl^-\rightarrow [Zn(Cl)_2], \quad (6)$$

$$[Zn(Cl)_2]+\text{Inhibitor}\rightarrow [Zn^{2+}\cdot\text{Inhibitor}]+2Cl^-. \quad (7)$$

Corrosion inhibitors of soursop leaf extract form a thin layer to protect the zinc surface from exposure to corrosion (this layer contains $[Zn(Cl)_2]$, $Zn(OH)_2$, $Zn_5(OH)_8Cl_2\cdot H_2O$, $[Zn^{2+}\cdot$Inhibitor$]$, etc.) in the corrosive medium NaCl (wet atmosphere corrosion), thus protecting zinc and inhibiting its corrosion.

Conclusions

In this study, the extraction of soursop leaves was conducted using the maceration method. The amount of extract obtained at the ratio of the weight of samples to the volume of solvents of 1:4 g/mL, 1:6 g/mL, 1:8 g/mL, and 1:10 g/mL were 1.52 g, 2.31 g, 3.34 g, and 4.17 g, respectively. The lowest zinc corrosion rate of 2.28 mpy and inhibitor efficiency of 61.20% on immersion in 3% NaCl medium was achieved at a ratio of sample to solvent of 1:10 with eight days of immersion. The results of this study provide information that alkaloid compounds from soursop leaves can be used as efficient inhibitors of the corrosion of zinc.

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ЕКСТРАКЦІЯ ЛИСТЯ СОУ-СЕПА (*ANNONA MURICATA* L.) ЯК ДЖЕРЕЛО ІНГІБІТОРУ КОРОЗІЇ ЦИНКУ В СЕРЕДОВИЩІ ХЛОРИДУ НАТРІЮ

Вікрам Александер, Ронданг Тамбун, Яманіель Гінтінг, Бунгаран Тамбун

На сьогодні, інгібітори корозії, одержані з органічних матеріалів, є переважаючими в промисловості, оскільки органічні інгібітори є екологічно чистими та дешевшими, ніж неорганічні інгібітори. Дане дослідження було спрямоване на визначення здатності екстракту листя соу-сепа (сметанне яблуко, Annona muricata L.) інгібувати корозію цинку. Екстракцію листя соу-сепа проводили методом мацерації з використанням етанолу як розчинника. У цьому дослідженні варіації у співвідношенні маси порошку листя соу-сепа до об'єму розчинника становили 1:4 г/мл, 1:6 г/мл, 1:8 г/мл і 1:10 г/мл, а тривалість занурення становила дві доби. Одержаний екстракт досліджували як інгібітор корозії цинку з використанням 3%-ного розчину хлориду натрію як імерсійного середовища. Концентрація інгібітору становила 2%, тривалість занурення варіювалася від 2 до 8 діб. Отримані результати показали, що при співвідношенні маси порошку листя соу-сепа до об'єму розчинника 1:10 найнижча швидкість корозії (2,28 mpy) та ефективність інгібування (61,20%) спостерігалися після восьми днів занурення.

Ключові слова: алкалоїд; мацерація; листя соу-сепа; корозія; органічний інгібітор.

EXTRACTION OF SOURSOP LEAVES (*ANNONA MURICATA* L.) AS A SOURCE OF ZINC CORROSION INHIBITOR IN SODIUM CHLORIDE MEDIUM

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Keywords: alkaloid; maceration; soursop leaf; corrosion; organic inhibitor.

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