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Synthesis of Schiff Bases via Condensation of Aldehydes from Ozonolysis of Purple Passion Fruit Seed Oil with Phenylhydrazine and Aniline and Their Utilization as Corrosion Inhibitor on Steel Metal (Zn) in 0.1N HCl Solution Media

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ABSTRACT

Schiff base was synthesized through a condensation reaction between the aldehyde derived from purple passion fruit seed oil and phenylhydrazine (Schiff base I) and the aldehyde derived from purple passion fruit seed oil with aniline (Schiff base II). Aldehydes are obtained from the ozonolysis process of purple passion fruit seed oil using ozone to produce ozonides and then reduced with Zn powder in dilute acetic acid. The formation of aldehyde derivatives of purple passion fruit seed oil is supported qualitatively by the formation of a brick red precipitate through the addition of Fehling reagent and the formation of a silver mirror on the wall of the test tube with the addition of Tollens reagent and a decrease in the number of iodine compared to purple passion fruit seed oil and the results of FT-IR analysis provide a spectrum with a vibration peak at a wave number of 1744.4 cm^{-1} which indicates the C=O aldehyde group. The aldehyde derivative of purple passion fruit seed oil produced has an iodine value of 110.0467, which is lower than that of purple passion fruit seed oil, which is 122.0644. The formation of Schiff II base resulted in yields of 3% and 70% supported by FT-IR spectrum of vibration peaks at wave numbers 1654 cm-1 and 1602.8 cm^{-1}, which indicated the formation of imines (-C=N-) as a result of condensation between derived aldehyde groups of purple passion fruit seed oil with amine groups of phenylhydrazine and aniline. Testing the iodine number on the Schiff I base and the Schiff II base yielded values of 124.9079 and 117.3266. A corrosion inhibitor test was carried out using the gravimetric method on HCl media with a concentration of 0.1N, 24 hours and various concentrations of compounds 1000, 3000, 5000, and 7000 ppm. Corrosion inhibitor efficiency for purple passion fruit seed oil, Schiff Base I, and Schiff Base II, respectively, were 27.87%, 88.28% and 94.08% at a concentration of 7000 ppm.

Keywords: Aniline, Corrosion Inhibitor, Phenylhydrazine, Purple Passion Fruit Oil, Schiff's Base

ABSTRAK

Basa Schiff disintesis melalui reaksi kondensasi antara aldehida turunan minyak biji markisa ungu dengan fenilhidrazin (basa Schiff I) maupun aldehida turunan minyak biji markisa ungu dengan anilin (basa Schiff II). Aldehida diperoleh dari proses ozonolisis minyak biji markisa ungu menggunakan ozon menghasilkan ozonida lalu direduksi dengan serbuk Zn dalam asam asetat encer. Terbentuknya aldehida turunan minyak biji markisa ungu yang secara kualitatif didukung dengan terbentuknya endapan merah bata melalui penambahan pereaksi Fehling dan terbentuknya endapan cermin perak pada dinding tabung reaksi dengan penambahan pereaksi Tollens serta menurunnya bilangan iodin dibandingkan terhadap minyak biji markisa ungu dan hasil analisis FT-IR memberikan spektrum dengan puncak vibrasi pada bilangan gelombang 1744,4 cm^{-1} yang menunjukkan gugus C=O aldehida. Aldehida turunan minyak biji markisa ungu yang dihasilkan memiliki bilangan iodin sebesar 110,0467 yang lebih rendah dibandingkan terhadap minyak biji markisa ungu yaitu sebesar 122,0644. Terbentuknya basa Schiff I dan basa Schiff II menghasilkan rendemen sebesar 73% dan 70% didukung melalui hasil analisis FT-IR dengan spectrum puncak vibrasi pada bilangan gelombang 1654 cm-1 dan 1602,8 cm^{-1} yang menunjukkan terbentuknya ikatan imina (-C=N-) hasil kondensasi antara gugus aldehida turunan minyak biji markisa ungu dengan gugus amin dari fenilhidrazin dan anilin. Pengujian bilangan iodin pada basa Schiff I dan basa Schiff II menghasilkan nilai 124,9079 dan 117,3266. Uji inhibitor korosi dilakukan dengan metode gravimetri pada media asam HCL konsentrasi 0,1 N,waktu 24 jam dan variasi konsentrasi senyawa 1000, 3000, 5000, dan 7000 ppm. Efisiensi inhibitor korosi untuk Minyak biji markisa ungu,Basa Schiff I, Basa Schiff II secara berturut turut adalah 27,87%, 88,28%, dan 94,08% pada konsentrasi 7000 ppm.

Kata Kunci: Anilin, Basa Schiff, Fenilhidrazin Inhibitor Korosi, Minyak Biji Markisa Ungu

1. Introduction

Indonesia is one of the highest producers of horticultural crops, both fruit and vegetable crops. One of the local fruits expected to become a mainstay fruit is passion fruit. Purple is the most widely cultivated type of passion fruit in Indonesia (*Passiflora edulis*). Passion fruit is one of Indonesia's agricultural products with considerable production [1].

Passion fruit production in North Sumatra amounted to 8,379 tons in 2019, while in 2022, it amounted to 9,329 tons [2]. The center of purple passion fruit production in North Sumatra is Karo Regency, which, from 2005 to 2009, on average, produced 63.5 per cent per year of the total passion fruit produced in North Sumatra [3]. In this case, passion fruit contains saturated fatty acids in the form of palmitic acid 6.7%, stearic acid 1.76%, unsaturated fatty acids in the form of oleic acid 19.0%, linoleic acid 59.9%, and linolenic acid 5.4%. However, the interest of Indonesian people in processing passion fruit seeds still needs to be higher. One of the modifications that can be made to purple passion fruit seed oil is ozonolysis (maximization by ozone), which has been widely used to determine the structure of unsaturated compounds because this reaction causes the degradation of large molecules into smaller molecules that can be identified. Oxidation of alkenes contained in unsaturated fatty acid chains by ozone will produce ozonides. If ozonide is reductively resolved, it will produce an aldehyde [4]. The resulting aldehyde can be further modified into a Schiff base. This compound is usually formed by reaction condensation between a primary amine compound and an aldehyde or ketone compound (Bell et al., 1963).

Schiff base compounds contain azomethin groups (-CH=N-), usually formed from condensation reactions of primary amine compounds with carbonyl groups from aldehyde and ketone compounds [5]. Schiff bases can be synthesized through condensation of primary amines with carbonyl compounds. Conventionally, Schiff bases can be synthesized by refluxing primary amines and aldehydes or ketones in an organic solvent and in the presence of a small amount of acid or base [6].

Corrosion is a process of material degradation caused by the influence of a corrosive environment, which can be in the form of gases, acidic or alkaline solutions, seawater, humid air and environments containing microbes. Corrosion events occur very complexly, involving several factors that work together simultaneously, including microbiological, chemical, electrochemical, metallurgical, and mechanical characteristics. In their use, Schiff bases are very effective as corrosion inhibitors against metals, which can spontaneously form a layer to protect the material or material and are environmentally friendly [7].

Previous researchers have utilized unsaturated fatty acids from vegetable oils as a source of aldehydes through ozonolysis reactions, including Schiff bases resulting from condensation between methyl oleic aldehyde with ethylenediamine and aniline as a source of primary amines where the test was used as a corrosion inhibitor against zinc metal in 0.1N HCl media which gave a corrosion inhibition efficiency value of 73.30% for ethylenediamine at a concentration of 7000 ppm and for aniline of 80.09% at a concentration of 7000. ppm [8]

Soybean oil-derived Schiff bases as a source of aldehydes have been used as corrosion inhibitors on soft steel in 2N HCl acid media where soybean oil contains unsaturated fatty acids, which are then ozonolyzed to produce soybean oil-derived aldehyde compounds followed by condensation reaction with benzylamine as a contributor to the primary amine group. This study shows that the corrosion of metals can be inhibited to prevent metal loss [9].

Sinulingga (2014) have conducted research on the synthesis of Schiff Bases through the condensation reaction of ethylenediamine with aldehyde from castor oil ozonolysis as a corrosion inhibitor against zinc metal. From this research, the efficiency of the Schiff base corrosion inhibitor produced against zinc metal in 0.1N HCl corrosive solution provides greater efficiency than castor oil, a mixture of castor oil aldehyde and ethylenediamine [10].

Likewise, the Schiff base is the result of condensation between aldehyde methyl ester of palm oil-derived fatty acids with aniline and phenylhydrazine as the primary amine source, which is tested as a corrosion inhibitor against zinc metal in 0.1N HCl media which gives a corrosion inhibition efficiency value of 85.44% for phenylhydrazine at a concentration of 7000 ppm and 82.38% for aniline at a concentration of 7000 ppm [11].

From the description above, researchers are interested in synthesizing Schiff bases by utilizing purple passion fruit seed oil, then ozonolysis followed by condensation reactions with two types of primary amine compounds with phenylhydrazine and aniline and testing the efficiency of Schiff bases obtained as corrosion inhibitors against zinc metal in 0.1N HCl media.

2. Materials and Methods

2.1. Equipment

The tools used in this research include: desiccator, spatula, glassware, Ozonisator; oven, hot plate stirrer, analytical balance; filter paper, Stative, clamps, Rotary Evaporator, Vacuum, magnetic bar, FT-IR spectrophotometer, and mass gas chromatography, zinc (Zn) metal.

2.2. Materials

The materials used in this study include purple passion fruit seed oil, n-hexane, Na_2SO_4 anhydrous, aqua dest, methanol, $Na_2S_2O_3$, KI, toluene, CH_3COOH, aniline, Phenylhydrazine, Fehling reagent, tollens, Zn powder, Wijs solution, cyclohexane, amylum indicator, HCl 37%, Ethanol.

2.3. Extraction of Purple Passion Fruit Seed Oil by Maceration

As much as 5 kg of purple passion fruit was cleaned, and the seeds were taken and dried. Then, purple passion fruit seeds were dried and mashed using a blender and 750 g of fine passion fruit seeds into a plastic bottle and macerated with 2.5 L of n-hexane for five days. Then filtered, the filtrate obtained was evaporated solvent with a rotary evaporator and added Na_2SO_4 anhydrous. It was then tested by gas chromatography, FT-IR Spectroscopic Analysis and determination of iodine number value.

2.4. Ozonolysis of Purple Passion Fruit Seed Oil

Put 200 mL of purple passion fruit seed oil into a 1000 mL Erlenmeyer glass, add 100 mL of methanol solvent and 100 mL of 5% KI and stir. The mixture was Ozonolyzed for 20 hours at an ambient temperature of ≤ 10C̊ with an Ozonator. The result of ozonolysis was then reduced by adding 5 g of Zn powder and 200 ml of dilute acetic acid, stirred for ± 15 minutes. Zn was separated by filtration, and acetic acid was separated by washing with distilled water and then evaporated by vacuum distillation so that the residue was obtained as a mixture of aldehydes derived from purple passion fruit seed oil, then tested with Fehling reagent and Tollens reagent, and continued with FT-IR Spectroscopy analysis and determination of iodine number value following the Wijs method.

2.5. Synthesis of Schiff Base I From Aldehyde Derived from Purple Passion Fruit Seed Oil with Phenylhydrazine

A total of 10 g of purple passion fruit seed oil-derived aldehyde mixture (0.0106 mol) was dissolved in 20 ml toluene and put into a 250 ml triple neck flask. A reflux device was equipped with a water trap and thermometer. A stirrer was inserted, and 11.5 g of phenylhydrazine (0.106 mol) was slowly poured through a dropper funnel and refluxed for 5 hours at 115-120 C. The solvent and excess phenylhydrazine were evaporated by vacuum distillation. The results obtained were then tested for iodine number and analyzed with an FT-IR spectrophotometer, followed by a corrosion inhibitor efficiency test.

2.6. Synthesis of Schiff Base II from Aldehydes Derived from Purple Passion Fruit Seed Oil with Aniline

A total of 10 g of passion fruit seed oil-derived aldehyde mixture (0.0106 mol) was dissolved in 20 ml toluene and put into a 250 ml triple neck flask. A reflux device was set up, equipped with a water trap and thermometer. A stirrer was inserted, and 9.8 g of aniline (0.106 mol) was slowly poured through a dropper funnel and refluxed for 5 hours at 115-120C. The solvent and excess aniline were evaporated by vacuum distillation. The result was then tested for iodine number and analyzed by FT-IR spectrophotometer followed by corrosion inhibitor efficiency test.

3. Results and Discussion

3.1. Purple Passion Fruit Seed Oil Extraction

The oil obtained from the extraction of purple passion fruit seeds using n-hexane solvent through the maceration method, which produces a yield of 35.7% from 750 g of purple passion fruit seeds, obtained 267.75 g of oil with an iodine number value of 122.0644. Based on gas chromatography analysis, the content of unsaturated fatty acids contained in purple passion fruit seed oil is oleic acid at 9.874%, linoleic acid at 79.9503%, and palmitic acid at 7.8950%.

3.2. Ozonolysis of Purple Passion Fruit Seed Oil

Ozonolysis of purple passion fruit seed oil at ≤10 C̊ for 20 hours can produce aldehyde compounds in the form of a mixture of rubber seed oil-derived aldehydes. The addition of KI in the ozonolysis process aims to trap ozone first or remove the ambient ozone level (free ozone) in the ozonolysis process (Fick, 2003). The end of the ozonolysis process is to produce a change in color from brownish yellow to pale yellow, followed by the presence of foam appearing in the upper rim layer on the Erlenmeyer glass wall, which indicates that the ozonolysis reaction has been completed. The appearance of foam at the end of the ozonolysis process is due to the reaction between O_3 and KI to form KOH so that some ester compounds undergo saponification, followed by the addition of acid during reduction to produce carboxylic acids and then continue with the addition of Zn metal in the form of powder in dilute acetic acid (CH_3COOH 20%) which will reduce ozonide into a mixture of aldehydes derived from purple passion fruit seed oil. From 200 mL of purple passion fruit seed oil that was ozonolyzed, 68.5 ml (34.5%) of aldehyde mixture was obtained.

The test of Fehling's reagent, which produces a brick red precipitate and with Tollens reagent, which has a silver mirror precipitate on the test tube wall, shows a positive test for the aldehyde from ozonolysis. The iodine number in the aldehyde mixture derived from purple passion fruit seed oil is 110.0467, while in purple passion fruit seed oil, the iodine value obtained is 122.0644, which indicates that there has been a break in the π bond of unsaturated fatty acids contained in purple passion fruit seed oil. The hypothesized formation reaction of purple passion fruit seed oil-derived aldehyde mixture is shown in Figure 1.

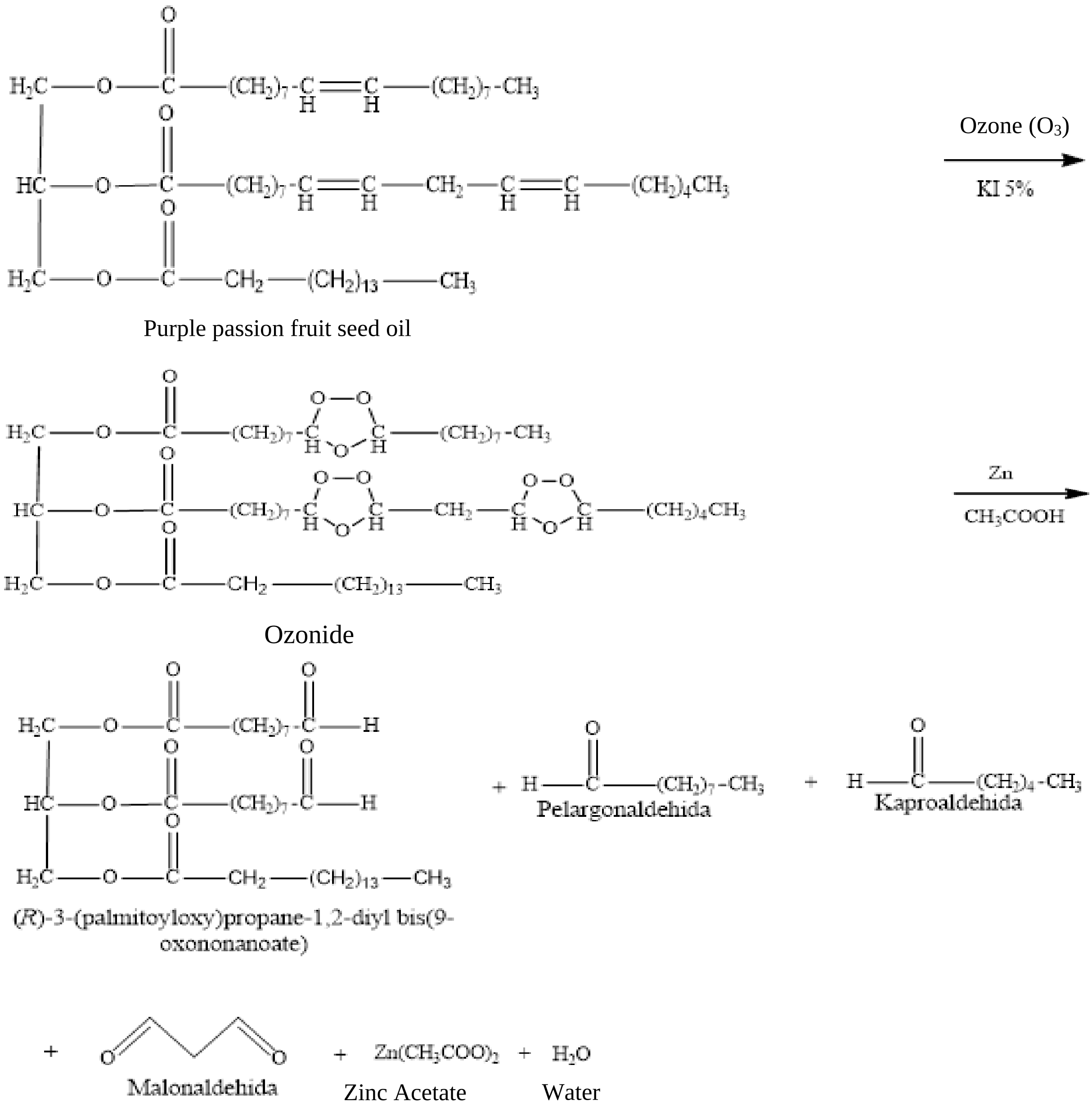

Figure 1: Formation reaction of aldehyde derivatives of passion fruit seed oil

3.3. Synthesis of Schiff Base I from A Mixture Of Aldehydes Derived from Passion Fruit Seed Oil with Phenylhydrazine

Schiff bases are produced from the condensation reaction of a mixture of aldehydes derived from purple passion fruit seed oil with phenylhydrazine by refluxing at 115-120ºC in toluene solvent for 5 hours so that a Schiff base bond (C=N) is formed between the C atom of the aldehyde group of the purple passion fruit seed oil-derived aldehyde mixture compound with the N atom of phenylhydrazine. The reaction between the aldehyde mixture of purple passion fruit seed oil derivatives and phenylhydrazine begins with the attack of the N atom of phenylhydrazine on the aldehyde C atom of the aldehyde mixture of purple passion fruit seed oil derivatives and the aldehyde O atom attacks the H atom of phenylhydrazine. Then the O atom attacks 1 more H atom and separates to produce water so that the formation of a C=N double bond occurs and produces Schiff base I. A Schiff base I produced as much as 13.7551 g and obtained a yield of 73% from 10 g of aldehyde mixture and 11.5 g of phenylhydrazine, where this weight was weighed after the vacuum distillation process to separate the remaining phenylhydrazine due to excessive addition at the beginning of the reaction. The addition of excess phenylhydrazine is intended so that the primary amine group on phenylhydrazine can completely condense the carbonyl group in the aldehyde mixture compound. The increase in iodine number from 110.0567 to a mixture of aldehydes derived from purple passion fruit seed oil to 124.8479 to Schiff base I indicates that a C=N bond has been formed on the Schiff base I. The hypothesized reaction formed can be seen in Figure 2.

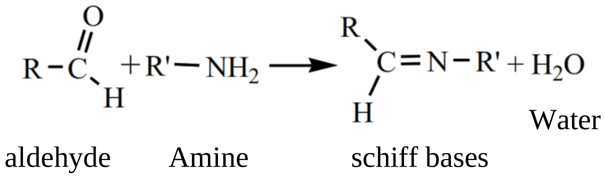

Figure 2: Schiff Bases Reactions

Figure 2 Description:
R'= $-(CH_2)_7-CH=N-NH-C_6H_{12}$ (Schiff bases I),
R'= $-(CH_2)_7-CH=N-C_6H_{12}$ (Schiff bases II)

3.4. Synthesis of Schiff Base II from a Mixture of Aldehydes Derived from Passion Fruit Seed Oil with Aniline

Schiff bases were synthesized from the condensation reaction between a mixture of aldehydes derived from purple passion fruit seed oil with aniline as a primary amine source in toluene solvent refluxed for 5 hours so that a Schiff base bond (C=N) was formed between the C atom of the aldehyde group of the purple passion fruit seed oil-derived aldehyde mixture with the N atom of aniline. The reaction between the aldehyde mixture of purple passion fruit seed oil derivatives with aniline begins with the attack of the N atom of aniline on the aldehyde C atom of the aldehyde mixture of purple passion fruit seed oil derivatives, and the aldehyde O atom attacks the H atom of aniline [12]. Furthermore, the aldehyde O atom attacks 1 more H atom and separates to produce water, and double bond formation occurs, which produces Schiff base II. The resulting Schiff II base of 12.4491 g obtained a yield of 70% from 10 g of aldehyde mixture and 9.8 g of aniline, where this weight was obtained after the vacuum distillation process to separate the remaining excess aniline. The addition of excess aniline in the initial reaction ensures that the carbonyl group in the aldehyde mixture compound can be condensed entirely by the primary amine group in aniline [13]. The increase that occurs in the iodine number from 110.0467 to the rubber seed oil-derived aldehyde mixture to 117.2098 on the Schiff II base shows that there is a C=N bond on the Schiff II base.

3.5. FT-IR Analysis Results

Comparison of FT-IR spectra of purple passion fruit seed oil, mixed aldehyde derivatives of purple passion fruit seed oil, Schiff base I and Schiff base II showed changes in purple passion fruit seed oil compounds into a mixture of aldehyde derivatives of purple passion fruit seed oil characterized by the change of symmetrical C=O ester into unsymmetrical C=O aldehyde at wave number 1744.4 cm^{-1} and proven by qualitative tests using Fehling which produced a brick red precipitate and using Tollens produced a silver mirror precipitate. Furthermore, the appearance of C=N Schiff base I and C=N Schiff base II groups at wave numbers 1654.9 cm-1 and 1602.8 cm^{-1} [14]. The FT-IR spectrum of purple passion fruit oil seed oil changes into a mixture of aldehydes derived from purple passion fruit oil seed oil, which in turn becomes Schiff base I and Schiff base II, as seen in Figure 3.

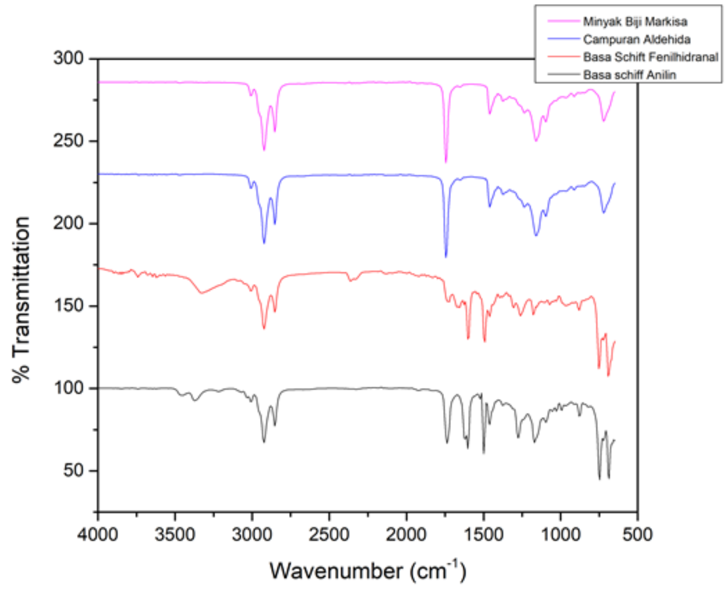

Figure 3: FT-IR spectra of passion fruit seed oil, the mixture of aldehydes derived from passion fruit seed oil, Schiff base I and Schiff base II

3.6. Determination of Corrosion Inhibitor Efficiency

Testing the efficiency of corrosion inhibitors was carried out by immersing zinc plates in 0.1 N HCl corrosion media solution with the use of inhibitors, namely purple passion fruit seed oil, Schiff Base 1, and Schiff Base 2, and without the use of inhibitors. The time variation used is 24 hours. As for the variation of inhibitor concentrations, they are 1000 ppm, 3000 ppm, 5000 ppm, and 7000 ppm.

Determination of corrosion inhibitor efficiency was carried out by immersion in 0.1N HCl media for 24 hours with variations in inhibitor concentrations of 1000 ppm, 3000 ppm, 5000 ppm, and 7000 ppm. Inhibitor efficiency testing is carried out on zinc metal because zinc metal is an active metal and has many applications in industry [15]. The average inhibitor efficiency values of the various samples can be seen in the table below:

Table 1: Immersion results of zinc plates without the use of inhibitors in 0.1 N HCl acidic media solution

Corrosion Inhibitor Concentration (ppm)	Soaking Time (hour)	Initial weight of zinc plate (g)	Final weight of zinc plate (g)	Zinc plate weight loss (g)	Inhibitor Efficiency (%)
0	24	2,4562	2,3666	0,0896	0

Table 2: Immersion results of zinc plates using purple passion fruit seed oil inhibitor in 0.1 N HCl acid media solution

Corrosion Inhibitor Concentration (ppm)	Soaking Time (hour)	Initial weight of zinc plate (g)	Final weight of zinc plate (g)	Zinc plate weight loss (g)	Inhibitor Efficiency (%)
1000	24	3.5956	3.5193	0.0763	14.84
3000	24	3.9131	3.8406	0.0725	19.08
5000	24	3.5734	3.5034	0.0700	24.51
7000	24	3.6103	3.541	0.0693	27.87

Table 3: Immersion results of zinc plates using Schiff Base 1 inhibitor in 0.1N HCl acidic media solution

Corrosion Inhibitor Concentration (ppm)	Soaking Time (hour)	Initial weight of zinc plate (g)	Final weight of zinc plate (g)	Zinc plate weight loss (g)	Inhibitor Efficiency (%)
1000	24	3.8128	3.7832	0.0296	66.94
3000	24	3.7048	3.6853	0.0195	78.23
5000	24	3.8757	3.8597	0.0160	82.14
7000	24	3.8114	3.8023	0.0091	89.84

Table 4: Immersion results of zinc plates using Schiff Base 2 inhibitor in 0.1 N HCl acidic media solution

Corrosion Inhibitor Concentration (ppm)	Soaking Time (hour)	Initial weight of the plate zinc (g)	Final weight of plate zinc (g)	Plate weight loss zinc (g)	Inhibitor Efficiency (%)
1000	24	2.4904	2.4195	0.0409	54.33
3000	24	3.0041	2.9701	0.0339	62.16
5000	24	2.6005	2.5707	0.0298	74.76
7000	24	2.5262	2.5137	0.0125	86.09

The inhibitor compounds used in this research are purple passion fruit seed oil, Schiff base 1, and Schiff base 2. The three compounds have active properties as corrosion inhibitors. The efficiency of an inhibitor increases in line with the increasing concentration of inhibitors added to 0.1N HCl corrosion media. From the data obtained, the highest inhibitor efficiency is in Schiff Base 1 because there are more free electron pairs in Schiff Base I that can limit the diffusion of O_2 on the metal surface. The source of free electrons in Schiff Base I is the double bond (π bond) on benzene, the π bond on C = N and from N atoms. Whereas in Schiff Base II, the source of free electrons is less than in Schiff Base I. Wang (2023) states that the increase in the inhibition efficiency of inhibitors can be explained based on the adsorption theory, which considers that inhibitors form a layer/film This is due to the π electrons and free electron pairs on the N atoms that interact with the orbitals of the metal (Zn) forming a protective layer/film that can inhibit metal dissolution, so that the corrosion rate drops and the inhibition efficiency rises [16].

4. Conclusion

Purple passion fruit seed oil obtained 35.7%, and the ozonolysis process of purple passion fruit seed oil-derived aldehyde mixture got 68.5 mL (34.2%) with a decrease in iodine number from 122.0644 for purple passion fruit seed oil to 110.0467. Schiff base I of 13.7551 g obtained a yield of 73% with an iodine number value of 124.8979, and Schiff base II of 12.4991 g got a yield of 70% with an iodine number value of 117.2098 The formation of Schiff bases I and II is supported by the appearance of vibrational peaks in the wave number region of 1654.9 cm-1 and 1602.8 cm-1 respectively which indicates the C = N group. Next, the test of corrosion inhibitor efficiency against zinc metal in 0.1N HCl corrosive media was carried out by gravimetric method, immersion time of 24 hours and variation of compound concentration of 1000, 3000, 5000, 7000 ppm. The most outstanding corrosion inhibitor efficiency for Schiff base I is 89.84%, while for purple passion fruit seed oil and Schiff base II, it is 27.87% and 86.09% at a concentration of 7000 ppm. Thus, Schiff Base I is the best use of inhibitors as corrosion inhibitors against zinc metal.

5. Acknowledgments

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6. Conflict of Interest

The authors declare no conflicts of interest.

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