Corrosion Inhibitors Activity of Schiff Base from Condensation of Ethylenediamine with Furfural from Sugarcane Bagasse

Mimpin Ginting, Dasron Bulolo, Herlince Sihotang and Indra Masmur
Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sumatera Utara, Medan, Indonesia

Keywords: Schiff Base, Ethylenediamine, Furfural, Corrosion Inhibitor, Zinc.

Abstract: Schiff Base from condensation of ethylenediamine as the source of primary amine with furfural from sugarcane bagasse as the source of carbonyl has been synthesized and tested for its corrosion inhibitors activity. The yield of furfural from 100 g of sugarcane bagasse is 12.6 g. Qualitative analysis shows the existence of furfural in brick red colour by using aniline : acetic acid (1:1 v/v) as solvent. FT-IR Spectroscopy shows vibration in wavenumber 1677 cm^{-1} as C=O and 1573 cm^{-1} as cyclic C=C. Condensation 9.6 g of furfural with 2.4 g of ethylenediamine in reflux condition for 5 hours using ethanol as solvent yields 8.64 g (70.55%) of Schiff Base. FT-IR Spectroscopy for Schiff Base analysis shows vibration of –C=N- in 1647 cm^{-1}. The test of corrosion inhibitor activity is by weighting the decrease mass of zinc in HCl 0.1 N with concentration 7000 ppm for each compound. The efficiency value of corrosion inhibitor are 69.06% for furfural, 53.67% for ethylenediamine, and 82.20% for synthesized Schiff Base.

1 INTRODUCTION

Sugarcane bagasse is a solid part of cane from extracted cane stem. Sugarcane bagasse consists of C (carbon) 47%, H (hydrogen) 6.5%, O (oxygen) 44%, and ash 2.5%. Based on Pritzelitz formula (Hugot, 1986) every kilogram of sugarcane bagasse contains 2.5% sugar which produce 1825 kcal/kg. Composition of sugarcane bagasse are 3.82% ash, 22.09% lignin, 37.65% cellulose, 27.97% pentosan, 3.01% silica, and 3.3% reduction sugar. Basically, fiber of sugarcane bagasse consists of cellulose, pentosan, and lignin. Composition of each component is vary depend on the variety of cane (Mubin & Ratnanto, 2005) Sugarcane bagasse also contains polysaccharides which can be converted into many industry production. One of polysaccharides components in sugarcane bagasse is pentosan (20-27%).

With high concentration, pentosan from sugarcane bagasse can be converted into furfural. Furfural has aldehyde carbonyl functional group which can be transformed into its derivatives like alcoholic furfuryl, furan, etc. Schiff Base is one of organic compounds which contain imine (-HC=N-) which can be synthesized from condensation of carbonyl group like aldehyde or ketone with primary amine (Cinerman et al., 1997). Schiff Base is one of organic compound with many uses. It can be use as pigment and dye, catalyst, intermediate in organic synthesize, and polymer stabilizer (Dhar & Thappo, 1982). Some of research have found that Schiff Base can be used as corrotion inhibitor for metal which spontaneously form a layer to protect materials and friendly environmentally (Li et al., 1999). Previous researchers have synthesized Schiff Base from cynnamaldehyde with 2-aminophenol as corrotion inhibitor for iron in HCl 0.5 N as media with inhibitor efficiency 92% (Qasim, 2011). Schiff Base from condensation of cynnamaldehyde as the source of carbonyl with ethylenediamine as the source of amine in concentration 7000 ppm for zinc in HCl 0.1N as media with inhibitor efficiency 90.17% (Ginting et al., 2016). Corrotion is a decrease quality of metal because electrochemical reaction with environment. (Trethewey and Chamberlain, 1991).Corrotion is a big problem for metal-formed material thing like car, bridge, ship machine, etc (Riegher, 1992). Metals can be broken and lost its function because of corrotion. Corrotion in many things can not be prevented but the rate of corrotion can be inhibited (Callister, 1991). Based on the descrioption, we interest to synthesize Schiff Base from furfural from sugarcane bagasse isolation wirhethylenediamine. The result obtained is expected has corrotion inhibitor activity and can be tested in rusty zinc with HCl 0.1 M as media.

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2 RESEARCH METHODOLOGY

2.1 Materials and Methods

Used equipments in this research are : condenser, hotplate stirrer, thermometer, vacuum distillation tools, analytical balance, UV-Vis Spectrophotometer, FT-IR Spectrophotometer, desiccators, and glasswares. Then used materials in this research are sulfuric acid (p.a), ethanol (p.a), ethylenediamine (p.a), furfural, zinc plate, aquadest, sugarcane bagasse, sodium chloride (p.a), and hidrocloric acid (p.a).

2.2 Isolation of Furfural from Sugarcane Bagasse

Put 100 grams of dried sugarcane bagasse in one-neck flask 250 ml then add 100 grams of NaCl and H_2SO_4 10% until sink and stir until homogeneous. Arrange distillation tools and reflux the mixture for 5 hours in 106°C. Furfural and water will be condensed to distillation flask then put the drop of liquid into Erlenmeyer which contain chloroform. Furfural will be soluted in chloroform and water will be separated in two layers (top layer is water and bottom layer is furfural/chloroform). The mixture of water and chloform is separated by dropping funnel. Then, add 1 gram Na_2SO_4anhidrous to chloroform layer to separate water and filter it. The filtrate then distillate in 61°C-65°C to evaporate chloroform. Furfural residue is purified by vacuum distillation where furfural is in destilate form in 130°C/8 mmHg. Identify the colour by using aniline acetate (1:1 v/v) as solvent, then analysed by UV-Vis Spectrophotometer and FT-IR Spectrophotometer.

2.3 Synthesize Schiff Base from Condensation of Furfural with Ethylenediamine

Put 9.6 grams (0.1 mol) of furfural in two neck flask 250 ml then soluted with 25 ml ethanol absolute. Arrange reflux tools with magnetic bar, thermometer, and water trap. Put 2,4 grams (0.04 mol) of ethylenediamine which soluted with 25 ml ethanol absolute and drop slowly by dropping funnel and stir with reflux condition for 5 hours. The solution then evaporate by rotaryevaporator, then excess furfural is evaporated by vacuum distillation. Then, dry residue from evaporation in desiccators and weight on analytical balance and analyse by FT-IR Spectroscopyparagraphs.

2.4 Determination of Inhibitor Efficiency

Soaking solution for zinc plate is taken from inhibitor solution 1000 ppm, put 150 ml in a glassware. Soak sanded zinc 5 cm x 1 cm in the solution for 24 hours. Take the plate from media, wash carefully, then dry for 5 minutes and weigh the final weight. Calculate inhibitor efficiency by using this formula (Chitra et al., 2010):

$$\%EI = \frac{W_0 - W_1}{W_0} \times 100\%$$

Where
EI = Inhibitor Efficiency
W_0 = The lost of weigh without inhibitor use
W_1 = Thelost of weigh with inhibitor use

As comparation (control) use solution without inhibitor. With the same procedure, do the same thing with corrotion inhibitor variation 3000 ppm, 5000 ppm, and 7000 ppm in 48, 72, 96, and 120 hours. Do the same procedure for furfural, ethylenediamine, and Schiff Base.

3 RESULT AND DISCUSSION

3.1 Isolation of Furfural

Isolation of furfural is by hydrolysis pentosan from sugarcane bagasse to pentose by HCl and dehydration pentose by sulfuric acid to form furfural. Qualitative analysis for furfural by use aniline acetate shows brick red colour. 100 g sugarcane bagasse yields 5.2 g (5.2%) furfural. HCl is produced by reaction between NaCl with excess H_2SO_4 then excess H_2SO_4 is used as dehydrator to form furfural (figure 1).

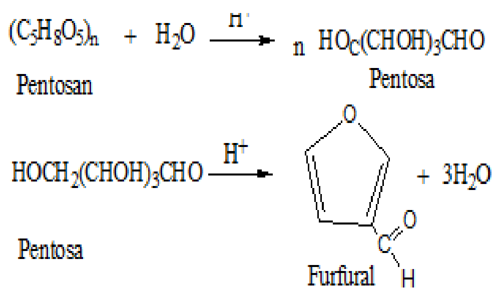

Figure 1: Furfural Formation Reaction.

UV-Vis spectroscopy analysis shows λmax in 273.1 nm which indicate furfural have conjugated diene bond (picture 2). FT-IR Spectroscopy result for furfural shows spectrum peak in 3100 cm^{-1} for stretching aromatic C-H, 2900-2800 cm^{-1} for stretching C-H aldehyde, 1740-1720 cm^{-1} for stretching C=O, 1600-1475 cm^{-1} for aromatic C=C, 1300-1000 cm^{-1} for stretching C-O-C, and 1500-1300 cm^{-1} for bending C-H aldehyde (figure 3).

3.2 Synthesize of Schiff Base (9*E*)-*N*[1],*N*bis((furan-2-yl)metil)etane-1,2-diamine from Condensation of Furfural with Ethylenediamine

Schiff Base is produced by condensation between furfural and ethylenediamine as the Schiff Base is produced by condensation between furfural and ethylenediamine as the source of primary amine with ethanol as the solvent reflux for 5 hours (picture 4). The product then purified by vacuum distillation. Condensation of 9.6 g (0.1 mol) furfural with 2.4 g (0.04 mol) ethylenediamine yields 8.64 g (70.55%) Schiff Base. Spectroscopy analysis for Schiff Base is indicated by vibration peak in 1647 cm^{-1} as –HC=N

3.3 Corrotion Inhibitor Efficiency Calculation

Inhibitor efficiency is tested by soaking zinc plate in corrotion media solvent HCl 0.1 N by using furfural, ethylenediamine, Schiff Base as inhibitor and media without inhibitor. Time variation are 24 hours, 48 hours, 72 hours, 96 hours, and 120 hours. Variation of inhibitor concentration are 1000 ppm, 3000 ppm, 5000 ppm, and 7000 ppm. Average inhibitor efficiency value can be look in table 1, 2, 3, and 4 source of primary amine with ethanol as the solvent reflux for 5 hours (picture 4). The product

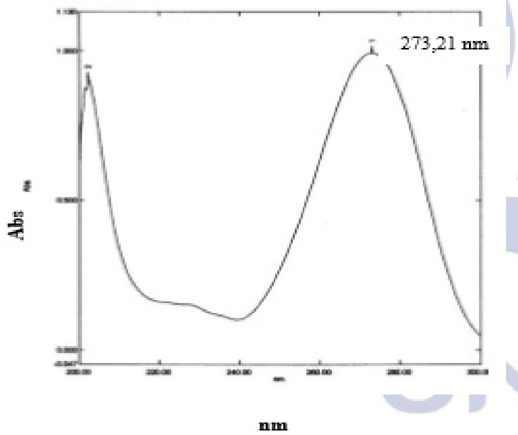

Figure 2: UV-Vis Spectrum for Isolated Furfural.

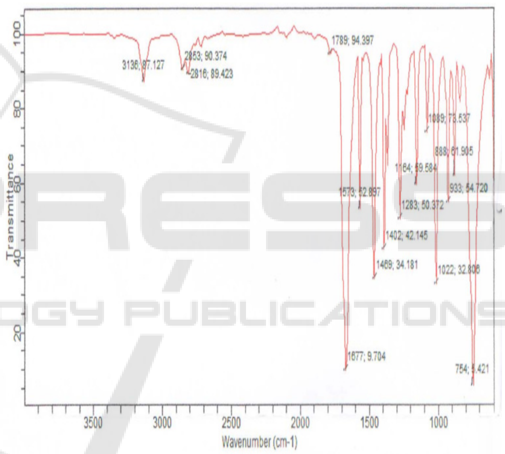

Figure 3: FT-IR Spectrum for Furfural.

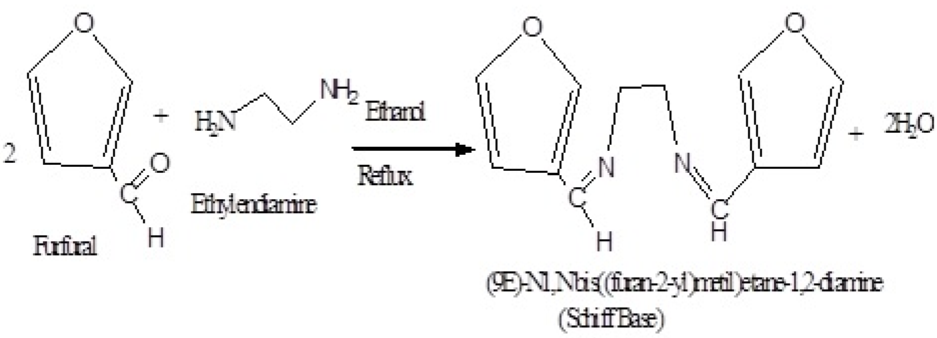

Figure 4. Formation and reaction of Schiff Base by condensation furfural with ethylenediamine.

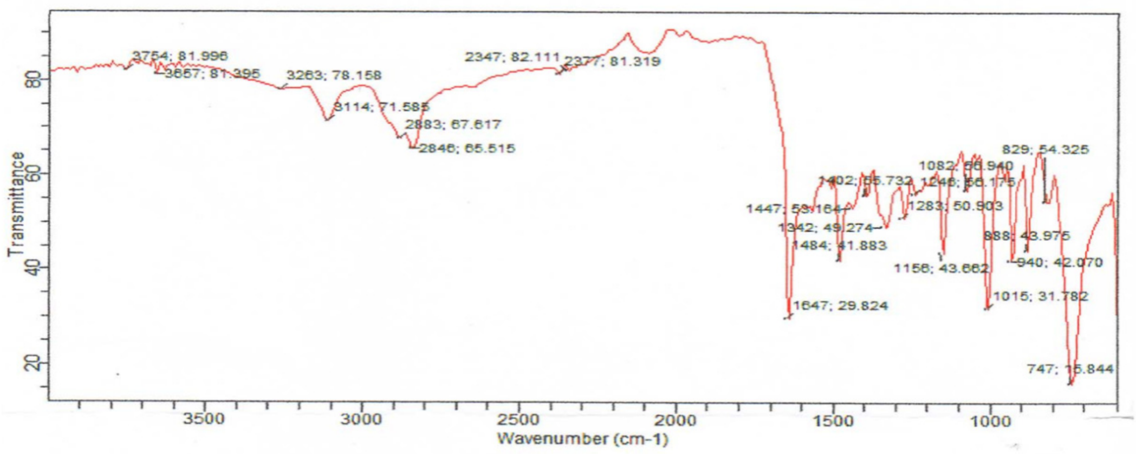

Figure 5: FT-IR Spectrum of Schiff Base (*9E*))-*N*[1],*N*[2]-bis((furan-2-yl)methyl)etane-1,2-diamine.

Table 1: Result of Zinc Plate Soaking without inhibitor in Corrosive Media Solution HCl 0.1 N.

Inhibitor Concentration (ppm)	Soaking Time (hours)	Zinc Initial Weight (g)	Zinc Final Weight (g)	Lost of Weight (g)	Inhibitor Efficiency (%)
0	24	2.9909	2.8811	0.1098	
0	48	3.3514	3.2309	0.1205	
0	72	3.2925	3.1623	0.1302	
0	96	3.2750	3.1047	0.1703	
0	120	3.2957	3.0613	0.2344	

Table 2: Result of Zinc Plate Soaking with Schiff Base (9*E*)-*N*[1],*N*[2]-bis((furan-2-yl)methyl)etane-1,2-diamine as inhibitor in Corrosive Media Solution HCl 0.1 N.

Inhibitor Concentration (ppm)	Soaking Time (hours)	Zinc Initial Weight (g)	Zinc Final Weight (g)	Lost of Weight (g)	Inhibitor Efficiency (%)	Average Inhibitor Efficiency (%)
1000	24	3.5031	3.4522	0.0509	53.64	49.48
	48	3.4221	3.3644	0.0577	7.38	
	72	3.3342	3.2548	0.0794	39.01	
	96	3.3021	3.2518	0.0503	70.46	
	120	3.2358	3.1817	0.0541	76.91	
3000	24	3.2133	3.2133	0.0381	68.38	59.71
	48	3.1106	3.0612	0.0494	55.00	
	72	3.1244	3.0580	0.0664	61.00	
	96	3.7525	3.6832	0.0693	70.43	
	120	2.4105	2.3373	0.0732	43.77	
5000	24	3.2777	3.2540	0.0237	71.41	77.88
	48	3.5149	3.4819	0.033	72.61	
	72	3.5167	3.4827	0.034	73.88	
	96	3.3860	3.3544	0.0316	81.44	
	120	3.5125	3.4729	0.0396	83.10	
7000	24	3.2530	3.2310	0.022	79.96	82.20
	48	3.6414	3.6119	0.0295	75.51	
	72	3.1121	3.0912	0.0209	83.94	
	96	3.5566	3.5214	0.0352	79.33	
	120	3.1347	3.1166	0.0181	92.27	

Table 3: Result of Zinc Plate Soaking with Furfural as Inhibitor in Corrosive Media Solution HCl0.1 N.

Inhibitor Concentration (ppm)	Soaking Time (hours)	Zinc Initial Weight(g)	Zinc Final Weight (g)	Lost of Weight (g)	Inhibitor Efficiency (%)	Average Inhibitor Efficiency (%)
1000	24	2.3305	2.2880	0.0425	61.29	45.60
	48	2.1419	2.0972	0.0447	60,90	
	72	3,5105	3.4533	0.0572	56.06	
	96	3.6501	3.5012	0.1489	12.56	
	120	3.4551	3.3033	0.1518	35.23	
3000	24	3.0941	3.0357	0.0584	46.81	51.10
	48	3.2933	3.2139	0.0794	34.10	
	72	3.3144	3.2519	0.0625	51.99	
	96	3.9606	2.8909	0.0697	59.07	
	120	3.5411	3.4557	0.0854	63.56	
5000	24	3.1454	3.0964	0.049	55.37	62.89
	48	3.1520	3.1012	0.0508	57.84	
	72	3.1358	3.0822	0.0536	58.83	
	96	3.1314	3.0757	0.0557	67.29	
	120	3.2515	3.1932	0.0583	75.12	
7000	24	3.2219	3.1844	0.0375	65.84	69.06
	48	3.2116	3.1737	0.0379	68.54	
	72	3.0415	3.3572	0.0443	65.97	
	96	3.1725	3.1240	0.0485	71.52	
	120	3.1443	3.0821	0.0622	73.46	

Table 4: Result of Zinc Plate Soaking with Ethylenediamine as Inhibitor in Corrosive Media Solution HCl 0.1 N.

Inhibitor Concentration (ppm)	Soaking Time (hours)	Zinc Initial Weight (g)	Zinc Final Weight (g)	Lost of Weight (g)	Inhibitor Efficiency (%)	Average InhibitorEfficiency (%)
1000	24	3.2418	3.1862	0.0556	49.36	40.35
	48	3.1776	3.1082	0.0694	42.40	
	72	3.5145	3.4373	0.0772	40.70	
	96	3.3858	3.3010	0.0848	50.20	
	120	3.3211	3.1315	0.1896	19.11	
3000	24	3.3116	3.2486	0.0630	42.62	43.88
	48	3.3355	3.2551	0.0804	33.27	
	72	3.3845	3.3143	0.0702	46.08	
	96	3.5643	3.4665	0.0978	42.57	
	120	3.3442	3.2385	0.1057	54.90	
5000	24	3.5110	3.4312	0.0789	28.14	49.02
	48	3.3651	3.3120	0.0531	55.93	
	72	3.2741	3.2114	0.0627	51.84	
	96	3.3315	3.2418	0.0897	47.32	
	120	3.1308	3.0415	0.0893	61.90	
7000	24	3.2182	3.1887	0.0295	73.13	53.67
	48	3.3888	3.2080	0.0808	32.94	
	72	3.4141	3.3215	0.0926	28.87	
	96	3.2388	3.1811	0.0577	66.11	
	120	3.2146	3.1380	0.0766	67.32	

3.4 Result of Determination Inhibitor Efficiency

Determination of corrotion inhibitor efficiency is by soaking zinc plate in HCl 0.1 N as media for 24, 48, 72, 96, and 120 hours with concentration variation 1000 ppm, 3000 ppm, 5000 ppm, and 7000 ppm. Efficiency inhibitor is tested in zinc because it is an active metal and always used in industry (Shah et al., 2011). The metal will be reduced and oxidized.

3.5 The Effect of Soaking Time to the Lost Weight of Zinc in Corrotion Media HCl 0.1 N

The effect of soaking time to the corrotion increase in HCl 0.1 N based on variation time 24, 48, 72, 96, and 120 hours is very high. It proves that hidrocloric acid solution is a corrosive media. The speed of corrotion for zinc is parallel with the length of soaking time (table 1).

3.6 The Effect of Inhibitor Concentration to the Lost Weight of Zinc in Corrosive Solution Media HCl 0.1 N

The increase of inhibitor concentration is parallel with the decrease of lost weight in zinc. It is because adsorption of inhibitor molecules in the surface of zinc platecan form shielding layer from free electron in atoms like O, N, and phi bonding which limit O_2 diffusion in zinc surface.

3.7 The Effect of Increasing Inhibitor Concentration to Inhibitor Efficiency in Zinc Plate with HCl 0.1 N as Corrotion Media

Inhibitor compounds in this research are furfural, ethylenediamine, and Schiff Base. The compounds are active as corrotion inhibitor. Inhibitor efficiency is increase parallel with the increase of inhibitor concentration in HCl 0.1 N as corrosive media.

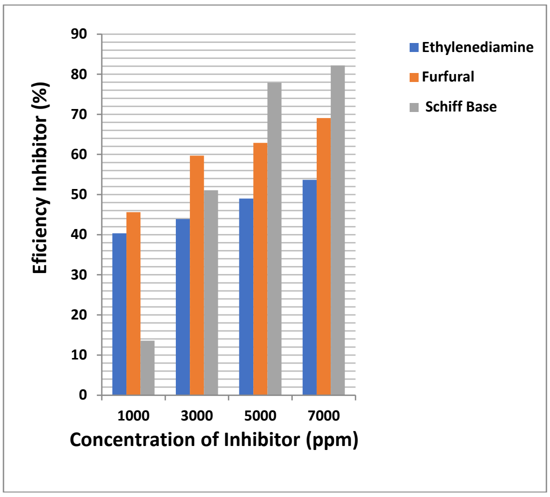

Figure 7: Graph of The Effect of Inhibitor Concentration to Inhibitor Efficiency.

4 CONCLUSSIONS AND SUGGESTIONS

4.1 Conclusions

Based on the research result and data analysis, the conclusions are:

1. Schiff Base can be synthesized by condensation ethylenediamine with furfural from sugarcane bagasse. The percentage of furfural from sugarcane bagasse is 5.2%. The rendemen of Schiff Base is 70.55%.
2. The result of Inhibitor efficiency test for zinc in HCl 0. 1 N as corrosive media with concentration 7000 ppm are 82.20% for Schiff base, 53.67% for ethylenediamine, and 69.06% for furfural.

4.2 Suggestions

The next researcher is expected to compare Schiff base efficiency from furfural with another primary amine source and the test of corrotion inhibitor in metal with many corrosive media.

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