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Synthesis of schiff base compound from vanillin and aniline with volume variations of acid catalyst from Belimbing Wuluh using grindstone method

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Abstract. Schiff base compound was a product between carbonyl and primary amine compounds in acidic conditions. Based on several studies, the compound had activity as a corrosion inhibitor. Schiff base compounds were synthesized from vanillin and aniline using natural acid catalysts from Belimbing Wuluh (*Averrhoa Blimbi* L.) with volume variations 0; 0.25; 0.5; and 1 mL and grinded for 10 minutes. The synthesized compound was characterized by its physical properties. Further characterization included a UV-Vis spectrophotometer, FTIR and GC-MS. The corrosion inhibition efficiency on metals was carried out in HCl 1M. Synthesized compounds were yellowish-white solids, slightly soluble in water, and had a melting point at 149-151°C. The yield with volume variations 0; 0.25; 0.5; and 1 mL in a row were 96.84; 93.81; 92.45 and 89.69%. The synthesized compound had wavelengths 283-284 and 325-330 nm. This compound had an imine bond (-C=N-) with wavenumber 1584.909 – 1585.974 cm^{-1}. The product with a 0 mL catalyst was characterized by GC-MS and showed a peak with retention time 24.173 minutes and m/z 227. It had a similarity with the molecular weight of the 2-methoxy-4-((phenylamino)methyl)phenol compound. The inhibition efficiency of these compounds was 39.38 to 77.40%.

1. Introduction

Schiff base compounds conventionally were synthesized by refluxing the mixture of reactant and organic solvent with the addition of a little bit acid catalyst. In common, the conventional method produced hazardous waste, took a long time and low yields [1-3]. Modification of the synthesis method was used to maximize the yield production while decreasing the production of waste and time. The modifications of the synthesis method were included the used of natural acids and the used of the grindstone method. The yields produced from the acid catalyst of mango juice were 71.14-91.11% and the yields from lime were 81.1-90.68% [4]. The grinding method produced yields with a range of 72-100% [5-7].

Schiff base compounds were produced by the reaction between the primary amine and the carbonyl group of the aldehyde or ketone. This compound had a characteristic of imine bonds (C=N). Characteristics of imine bonds in Schiff base compounds were obtained through the addition of a primary amine to carbonyl catalyzed by acids [8]. Schiff base compounds had many benefits in various sectors because of its unique structure. The utilization of these compounds included antifungal, antibacterial, anticancer, antitumor, anti-inflammatory, anti-insecticide, and antioxidant. Also, the Schiff base can be used as a corrosion inhibitor [1, 9-11].

2. Methods

Vanillin *p.a* and aniline *p.a* with a mol ratio of 1: 1 were mixed with natural acid catalyst from starfruit juice (*Averrhoa bilimbi L.)* and grinded using mortar and pestle for 10 minutes. The volume variation of acid catalyst were 0; 0.25; 0.5; and 1 mL. The product of synthesis observed physical properties including appearance, color, mass, and melting point. The solubility test was carried out with NaOH 2 M. Initial identification was carried out by TLC with the chloroform eluent. Further identification was made by UV-Vis, FTIR and KG-SM [1,5,7,11]. The best-synthesized results will be tested as corrosion inhibitors by the gravimetric method. The metals used were NT-Cutter BD-100 and the inhibitor solution was a mixture of HCl 1 M, DMSO, and the best-synthesized result. The variations of inhibitor were 1000, 3000, 5000, 7000 ppm. The corrosion inhibitor efficiency was calculated with the following equation 1 [1]:

$$\text{Yields} = \frac{Wo - W1}{Wo} \times 100\% \quad (1)$$

Wo = weight of metal without the addition of inhibitor
W1 = weight of metal with the addition of inhibitor

3. Result and discussion

3.1. Synthesis of Shiff base

The reaction between vanillin and aniline was illustrated in Figure 1. Schiff base compound 2-methoxy-4-((phenylamino)methyl)phenol was synthesized by the grindstone method and adding natural catalyst from Belimbing Wuluh juice. The process produced mechanical energy that is converted into heat energy. Heat energy would increase the activation of reactant molecules so that the collision process occurs and produce products. Acid catalysts played a role in the process of protonation during the elimination of H_2O. The physical properties of the synthesized compound were presented in Table 1.

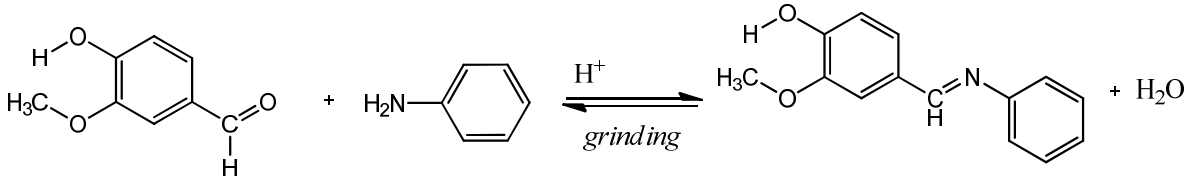

Figure 1. The scheme of Schiff base reaction.

Table 1. Physical properties of products.

Parameter	P1	P2	P3	P4
Physical form	Solid	Solid	Solid	Solid
Colour	Yellowish-white	Yellowish-white	Yellowish-white	Yellowish-white
Yields (%)	96,84	93,81	92,45	89,69%
Melting Point (°C)	150-151	149-150	150-151	149-151
Solubility	Slightly soluble	Slightly soluble	Slightly soluble	Slightly soluble

3.2. Characterization of product

3.2.1. *UV-Vis analysis.* The optimum absorption of spectrum form Schiff base 2-methoxy-4-((phenylamino)methyl)phenol was 283-284 and 325-330 nm.

3.2.2. *FTIR Analysis.* The unique characteristic of the Schiff base compound was it had imine bonds (C=N) in its structure. The imine functional group of synthesized compounds had strong absorption and were measured at 1584.909 – 1585.974 cm^{-1}. It was different compared with the spectra of vanillin and

aniline. Vanillin had a strong absorption of carbonyl (C=O) at 1665.841 cm^{-1}, while aniline had strong absorption of primary amine (N-H) at 3431.950 and 3356.057 cm^{-1}. FTIR spectra of synthesized compounds were showed in Figure 2.

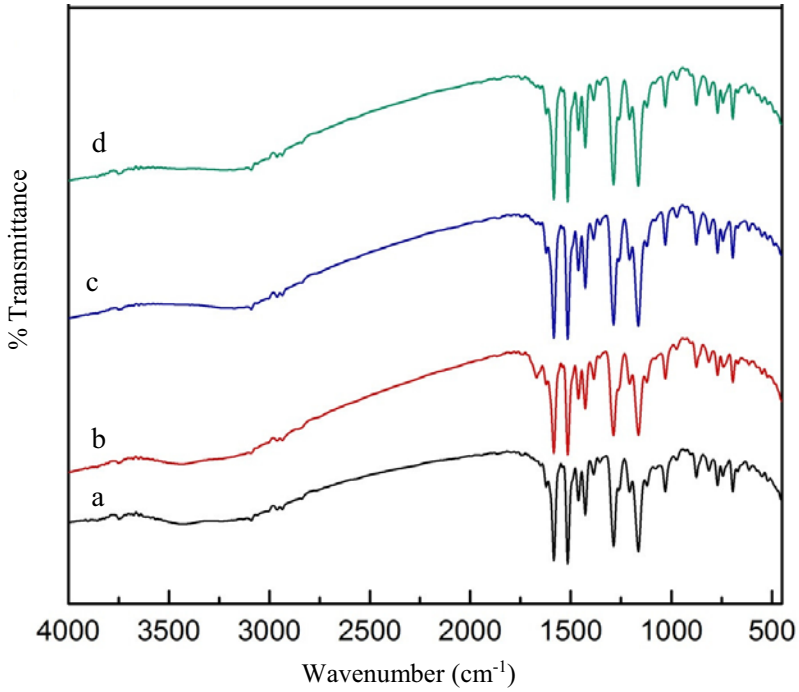

Figure 2. FTIR spectra of products with volume variations (a) 0, (b) 0.25, (c) 0.5, (d) 1 mL.

3.2.3. GC-MS Analysis. The analysis using GC-MS showed that the synthesized compound had high purity at 99.73%. The other small of it was the residual reactant. The product of synthesis had a molecular ion with m/z 227. It had a similarity with the molecular weight of Schiff base compound 2-methoxy-4-((phenylamino)methyl)phenol.

3.3. Corrosion inhibitor

Schiff base compounds effectively used as corrosion inhibitors because it had lone pairs of electrons on O and N atoms and had phi bonds (π) that would interact with the iron surface. Schiff base compounds would interact with ion-dipoles and create a layer on the surface of the iron that will coat and dispel the iron from corrosion. The efficiency of inhibitions was 39.38 to 77.40%.

4. Conclusion

The modification of the Schiff base synthesis method produced high yields with high purity. Furthermore, the synthesized product shows its activity as a corrosion inhibitor.

Acknowledgment

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