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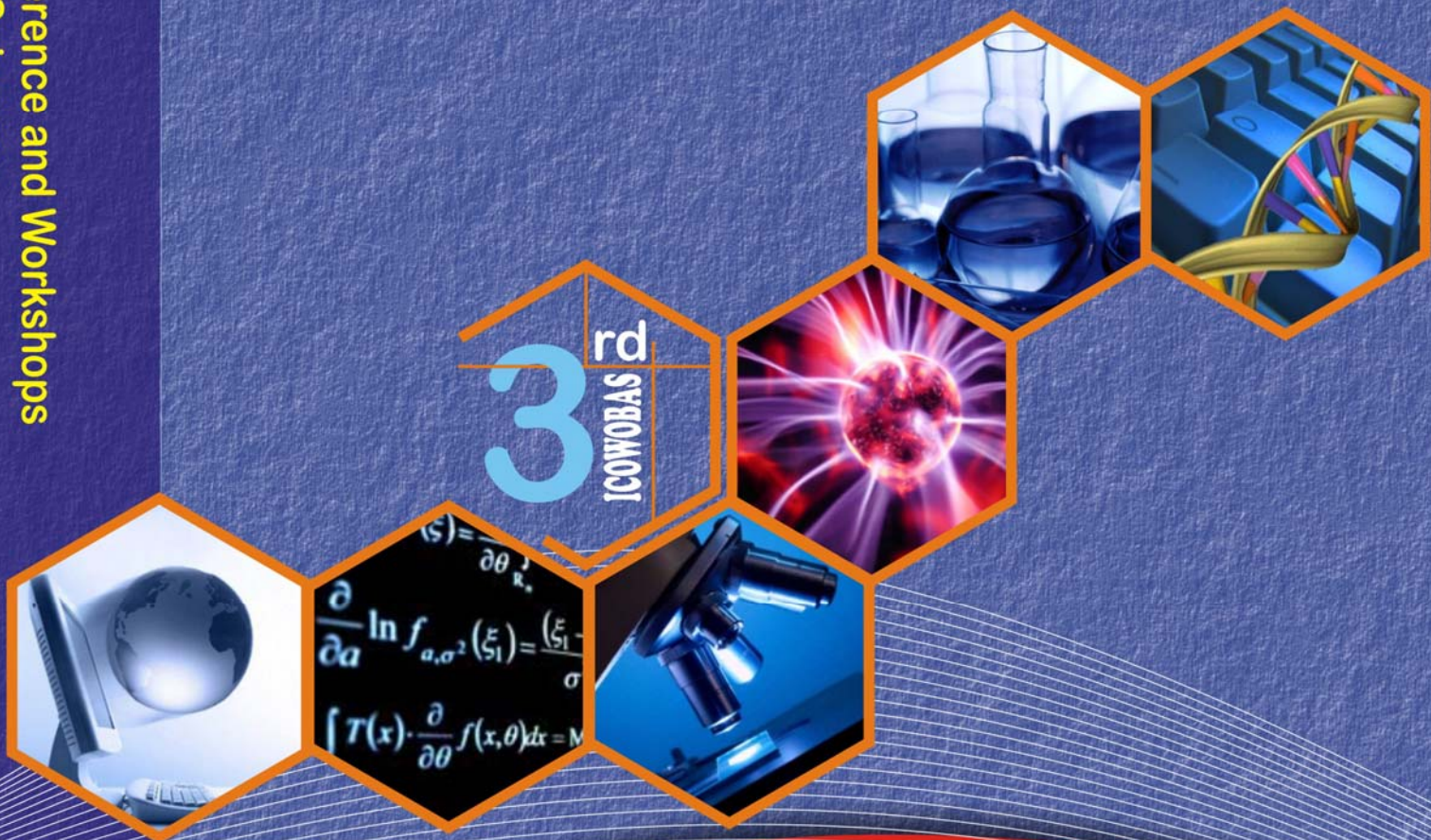
Proceedings

3rd International Conference and Workshops
on Basic and Applied Sciences

Enabling Research Innovation on Sciences
and Technology to Meet Global Challenges

Surabaya, Indonesia, September 21st - 23rd, 2011

Proceedings
3rd International Conference and Workshops
on Basic and Applied Sciences



UNAIR
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**ENABLING RESEARCH INNOVATION ON SCIENCES AND
TECHNOLOGY TO MEET GLOBAL CHALLENGES**

SEPTEMBER 21st - 23st

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Synthesis of Iron(III) Organometallic Compound

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Abstract

Ferroquine is an organo metallic compound, derivate of chloroquine and known of its more antimalaria activity than chloroquine. This organometallic structure model is used to design and synthesize another organometallic compound possessing antimalaria activity. In this research synthesis of [bis(2-(2,4-dimethoxybenzilidene)-6-methoxy-3,4-dihydro-2H-naphthalene-1-on)tetra(C₂H₅OH)Iron (III)] was done successfully. The ligand was synthesized by Claisen-Schmidt reaction of 6-methoxy-1-tetralone and 2,4-dimethoxybenzaldehyde. Organometallic compound was obtained by the reaction of the ligand with FeCl₃.6H₂O. Ligand and organometallic were characterized by spectroscopic method. The physical properties of the organometallic compound was determined by ESR and MSB.

Keywords: ferroquin, organometallic, antimalaria

1 Introduction

Malaria was caused by inoculation sporozoites in humans blood after the bite of an infected female *Anopheles* mosquito. About 40% of the world is malaria endemic areas. It now appears some new strains of malaria parasites resistant to antimalarial compounds. *Plasmodium sp* become resistant gradually to all anti-malarial compounds including chloroquine, pyrimethamine, sulfadoxine and halofantrine mefloquine (Domarle, 1998 and Nzila Alexis, 2006). Resistance is caused by mutation of *Plasmodium* genes, so that the antimalarial compounds were not able to inhibit of parasite growth anymore (Schlitzer, 2007).

Faced with this situation, it has found some new antimalarial design by developing the structure of the antimalarial know with the addition of metal into the chemical structure of the antimalarial like ferroquine (Pearson, 1985).

Ferroquine is one of antimalarial which its structure combined between chloroquine and ferrocene. The structures is figured below in Figure 1 (Pauson Peter L., 2001 and Daher, *et al.*, 2006).

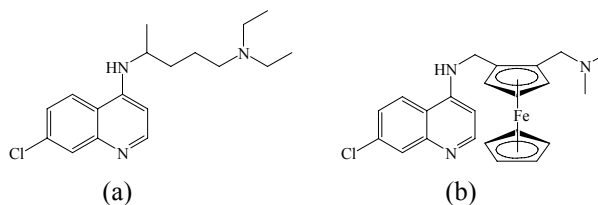


Figure 1: Chloroquine structure(a) dan ferroquine structure(b)

It was known that ferroquine had the IC₅₀ against *P. falciparum* with IC₅₀ of 0.78 nM while IC₅₀ of chloroquine was 1.9 nM. This suggested that ferroquine was 2.44 times more active than chloroquine. This data indicate that the use of organometallic compounds are very effective in lowering the resistance of *P. falciparum* (Atteke, *et al.*, 2003).

This research was done to the transform 2,4-dimethoxybenzaldehyde into an organometallic compound with a structure is resembling to ferroquine so it will hopefully also has an activity as antimalarial (Fitch, 1986 and Krogstad, 1987).

2 Methodology

2.1 Synthesis of Organometallic Iron(III)

Synthesis of ligand compound.

In a three neck round bottomed flask 0.5287 grams of 6-methoxy-3,4-dihydro-2H-naphthalene-1-one (3 mmol) was added, followed by 0.4980 grams of 2,4-dimethoxybenzaldehyde (3 mmol) in 9 mL ethanol. This mixture was refluxed at 5-10 °C and 3 ml of NaOH 40% (w/v) was added, while the temperature is maintained for 1 hour. Refluxed was continued for 4 hours at room temperature. The result of the reflux was cooled to form the precipitated product which then was filtered and recrystallized using ethanol (Kilway, 2007).

Determination of the maximum wavelength (λ_{max}) ligand 10^{-5} M.

About 10^{-5} M solution of ligand was placed in a cuvette and was measured the maximum wavelength at 190-350 nm.

Determination of stoichiometric Fe (III):ligand.

In 10 mL volumetric flasks, a solution of ligand with various concentrations was placed and was added with certain volume and certain concentration of a solution of Fe(III). Aquabides was added quantitatively. Each solution obtained was measured with a UV-VIS spectrophotometer. From this step the curve of ratio Fe(III):ligand mole against absorbance was obtained.

Synthesis of organometallic Iron (III).

$FeCl_3 \cdot 6H_2O$ and ligand were mixed using the ratio obtained from before step. This mixture was dissolved in 10 ml of ethanol and refluxed for 3 hours. Then the solution was heated until the color of third remaining solution was changed. The solution was cooled for several hours in order to form a perfect crystal. Furthermore, the crystal was filtered and washed repeatedly with ethanol. The crystals are dried at room temperature.

3 Result

3.1 2-(2,4-dimethoxybenzylidene)-6-methoxy-3,4-dihydro-2H-naphthalene-1-one

Yellow crystals, m.p. 110-112°C, yield = 72%, 1H NMR ($CDCl_3$, 600 MHz) ppm, (δ) = 2.890 (t, 2H, -CH₂-CH₂-), 3.026 (t, 2H, -CH₂-CH₂-), 3.836 (s, 9H, 3(-O-CH₃)), 6.502 (dd, 2H, Ar-H), 6.850 (dd, 1H, Ar-H), 7.231 (d, 1H, Ar-H), 7.948 (s, 1H, Ar-H), 8.105 (d, 1H, Ar-H) and 6.687 (s, 1H, =CH-). ^{13}C -NMR ($CDCl_3$, 600 MHz) ppm, (δ) = 186.93 (C=O), 163.50 (C-O), 161.73 (C-O), 159.85 (C-O), 145.85 (C_q), 134.12 (C_q), 132.20 (CH=CH), 131.09 (=CH-), 127.47 (C_q), 118.10 (C_q), 130.74 (=CH-), 113.28 (=CH-), 112.55 (=CH-), 104.39 (-CH₃), 98.27 (-CH₃), 55 (-CH₃), 29.60 (-CH₂-) and 27.85 (-CH₂-). FT-IR (KBr) cm^{-1} , 3063.01(=CH-), 1604.8 (C=C),

1504.5 (C=C aromatic) and 1458.21(C-H). MS m/z 325 [M+H]⁺.

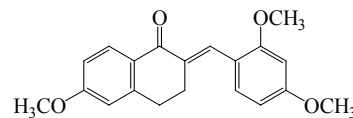


Figure 2: Ligand structure

3.2 [bis(2-(2,4-dimethoxybenzylidene)-6-methoxy-3,4-dihydro-2H-naphthalene-1-one)tetra(C₂H₅OH) iron(III)]

Brown crystals, m.p. 158-160°C, yield = 48%, FT-IR (KBr) cm^{-1} , 3425.04 (et-OH), 3063.01 (=CH-), 1604.8 (C=C), 1504.5 (C=C aromatic), 1458.21(CH) and 339.48 to 347.19 (C=O-Fe).

MSB (μ_{eff}) = 1 and ESR (Mn/MgO), g. 2.034-1.98 = 1. So it can be concluded that the organometallic compound has an unpaired electron and has a paramagnetic character.

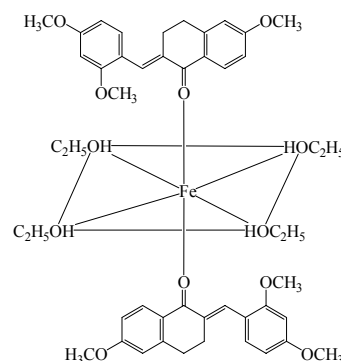


Figure 3: Organometallic structure

4 Conclusions

The result of research can be concluded that the compounds synthesized was [bis(2-(2,4-dimethoxybenzylidene)-6-methoxy-3,4-dihydro-2H-naphthalene-1-one)tetra(C₂H₅OH) iron(III)] and it will hopefully have activity as an antimalarial.

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