

SYNTHESIS, STRUCTURAL CHARACTERIZATION, BIOLOGICAL ACTIVITY OF COMPLEXES OF Mn(II), Cu(II) WITH 6-HYDROXY-3-SULFOQUINOL-7-YLOXYACETIC ACID

Nguyen Thi Ngoc Vinh¹

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Abstract: A novel quinoline derivative, 6-hydroxy-3-sulfoquinoline-7-yloxyacetic acid (**Q**), was successfully synthesized from eugenol, a major component of basil essential oil. Its corresponding complexes $[Mn(C_{11}H_7O_7NS)(H_2O)_2] \cdot H_2O$ (**B1**), $[Cu(C_{11}H_6O_7NS)(H_2O)_2] \cdot H_2O$ (**B2**) were obtained through direct coordination between Mn^{2+} , Cu^{2+} and ligand **Q**. The proposed structure of **B1**, **B2** was elucidated based on a comprehensive analysis of EDX, ESI-MS, IR spectroscopy, and thermal gravimetric data. In these complexes, the ligand **Q** coordinated to the Mn(II) center via the oxygen of OH phenol group, H_2O and O of COO group, resulting in a stable four-coordinate environment. The results of the antimicrobial activity test of the **B1** and **B2** complexes showed that both complexes had high activity against *Bacillus subtilis* and *Lactobacillus fermentum* strains, corresponding to low IC_{50} values ranging from 2.58 to 10.81 $\mu g/mL$.

Keywords: Mn(II), Cu(II) complexes, sulfoquinoline, structural, biological activity.

1. Introduction

The biological activity as well as anticancer and antimalarial properties of quinoline derivatives have been verified and have been used as therapeutic drugs. Quinoline compounds were widely used as antibiotics, fungicides, bactericides, dyes and flavoring agents. They were also used as catalysts, corrosion inhibitors and preservatives. Complexes of transition metals with quinoline derivatives often have various pharmacological properties such as anti-inflammatory, antibiotic, cytostatic, detoxifying, antibacterial and antimalarial activities [1-6].

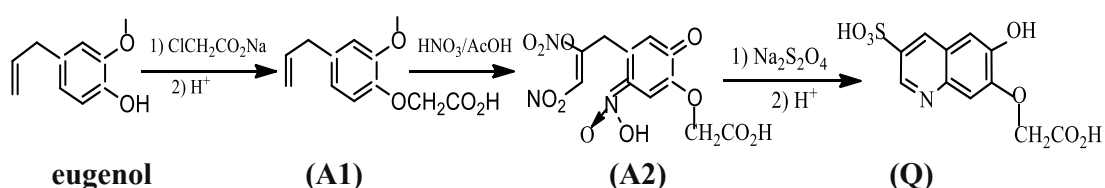
The sulfoquinoline derivative ligand named 6-hydroxy-3-sulfoquinol-7-yloxyacetic acid (denoted as **Q**) had been synthesized from eugenol found in basil essential oil [7]. This important compound had the ability to form complexes with transition metal ions via the OH group of phenol, the COOH group, ... The **B1** and **B2** complexes were synthesized, their structures were determined, and their biological activities were identified.

2. Materials and methods

2.1. Synthesis of **Q**

Q ligand was synthesized from eugenol in basil essential oil according to published procedure [7].

¹Faculty of Natural Sciences, Hong Duc University; E-mail: nguyenthingocvinh@hdu.edu.vn



2.2. Preparation of the complex B1

Added 1.5g of **Q** (5 mmol) to a glass beaker containing 10mL of DMF, stir until completely dissolved, slowly added 10ml of 0.5 M $MnCl_2$ solution (5 mmol) to the beaker, and stir the reaction mixture on a magnetic stirrer for 2 hours at room temperature. Added 40mL of acetone to the reaction mixture, and a yellowish-brown precipitate appeared. Filter the precipitate, wash with acetone, then dry at 45°C for 2 hours. Recrystallize the precipitate in an ethanol:water mixture of 1:1, cool in air, and obtain pale yellow crystals (**B1**). The reaction yield was 90%.

2.3. Preparation of complex B2

Added 1.5g of **Q** (5 mmol) to a glass beaker containing 10mL of DMSO stir until completely dissolved, slowly added 10mL of 0.5 M $CuCl_2$ solution (5 mmol) to the beaker, and stir the reaction mixture on a magnetic stirrer for 2 hours at room temperature. Added 40mL of acetone to the reaction mixture, and a yellowish-brown precipitate appeared. Filter the precipitate, wash with acetone, then dry at 50°C for 2 hours. Recrystallize the precipitate in an ethanol:water mixture of 1:1, cool in air, and obtain pale yellow crystals (**B2**). The reaction yield was 85%.

2.4. Exploration of biological activity

The antimicrobial activity of the complexes and ligand was tested at the Department of Applied Biochemistry - Institute of Chemistry, Vietnam Academy of Science and Technology.

3. Results and Discussion

Based on the results of spectral analysis methods, the molecular formulas of the complexes can be predicted to be $[Mn(C_{11}H_7O_7NS)(H_2O)_2] \cdot H_2O$ (**B1**), $[Cu(C_{11}H_6O_7NS)(H_2O)_2] \cdot H_2O$ (**B2**). In the +MS spectrum of complex **B1** (Figure 1), there was a cluster of ion peaks containing ion peaks with m/z value = 426.9 corresponding to ion $426.9 = \{[Mn(C_{11}H_7O_7NS)(H_2O)_2] + K^+\}^+$. Thus, the proposed molecular formula of complex **B1** was suitable.

Table 1. Result of elemental analysis (EDX), ESI-MS spectral of complexes

Numerical order	Complexes	Analysis results ESI-MS spectral	Result of EDX	
		Assignment (m/z)	Ratio atom (TN/LT) M : S	
1	B1	$426.9 = \{[Mn(C_{11}H_7O_7NS)(H_2O)_2] + K^+\}^+$	5,44 : 5,30/≈ 1:1	1:1
2	B2	$401.1 = \{[Cu(C_{11}H_6O_7NS)(H_2O)_2] + H^+\}^+$	3,84 : 3,99≈ 1:1	1:1

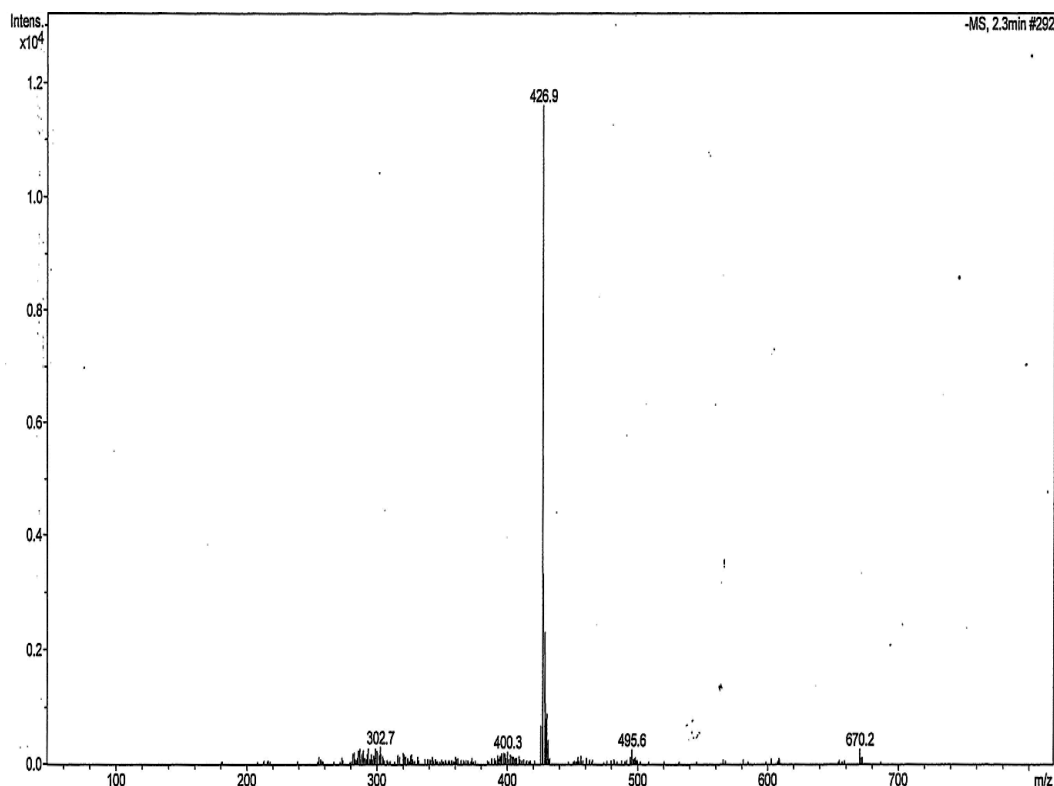


Figure 1. Result of the MS spectrum of B1 complex

In the IR spectra of complexes **B1** and **B2**, full absorption banded corresponding to the valence vibrations of the atomic groups in the molecule appeared. The main absorption bands were shown in Table 2.

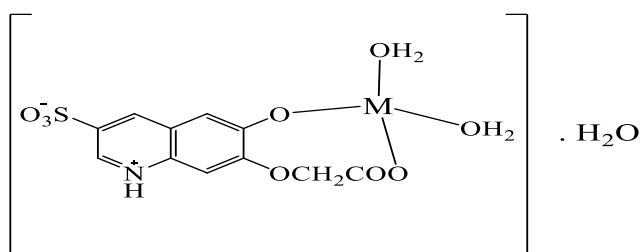
The results of thermal analysis of complexes **B1** and **B2** were presented in Table 1. On the thermographic analysis diagram of the **B1** complex with the formula: $[\text{Cu}(\text{C}_{11}\text{H}_6\text{O}_7\text{NS})(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$ at approximately 130 - 366°C, an endothermic effect appeared on the DTA curve and a decrease in mass on the TGA curve, this process was the loss of crystalline water and coordination water (Table 1). The solid remaining after decomposition of the complex at 800°C is expected to be MO. The mass percent of solid remaining calculated from the expected molecular formula was in good agreement with the mass percent of solid remaining calculated from the diagram.

Table 2. The main absorption bands on the infrared spectrum (cm^{-1}) and result of thermal analysis method of complexes and Q

Form name	The main absorption bands on the infrared spectrum (cm^{-1})				Result of thermal analysis method		
	ν_{OH}	$\nu_{\text{COO}^- \text{ kdx}}$	$\nu_{\text{COO}^- \text{ dx}}$	$\nu_{\text{C-O}}, \nu_{\text{C-N}}$	$\nu_{\text{M-O}}$	Dehydration n $\Delta m \%$, TN/LT	% MO After decompositi

						Crystallize and Coordinati on	on /Δm %, (TN/LT)
Q	3600-3200	1653		3600-3200			
B1	3600-3200	1595	1498	3600-3200	481	9.30/10.17	25.58/24.66
B2	3600-3200	1609	1493	3600-3200	512	12.36/11.3 2	33.18/32.22

Combining the results of analysis of EDX spectrum, ESI MS spectrum, IR spectrum, thermal analysis diagram, the structural formula of complexes **B1**, **B2** was proposed:



(M: Mn, Cu)

The results of the antimicrobial testing of the complexes and ligands showed that complexes **B1** and **B2** exhibited high activity on *Bacillus subtilis* and *Lactobacillus fermentum* with low IC₅₀ values from 2.58 ÷ 10.81 µg/mL (Table 3).

Table 3. Antimicrobial resistance test results of complexes

Numerical order	Form name	IC ₅₀ values for strains (µg/ml)				
		Gram (+)		Gram (-)		
		<i>Bacillus subtilis</i>	<i>Lactobacillus fermentum</i>	<i>Escherichia coli</i>	<i>Pseudomonas aeruginosa</i>	<i>Candida albican</i>
1	MeQBr	>128	>128	>128	>128	>128
2	CoMeQBr	2,45	0,86	75,62	>128	111,0
3	NiMeQBr	1,98	0,93	>128	>128	112,45

4. Conclusion

Two complexes **B1** and **B2** were synthesized by the reaction of Mn(II), Cu(II) with the ligand 6-hydroxy-3-sulfoquinol-7-yloxyacetic acid (**Q**). The structures of the complexes were determined by combining the results of IR, EDX, ESI-MS and thermal analysis spectra. In the complexes Mn(II), Cu(II) have a coordination number of 4. The central metal atom binds to the ligand through the oxygen of OH phenol group, H₂O and O of COO group.

The results of the antimicrobial testing of the complexes and ligands showed that complexes **B1** and **B2** exhibited high activity on *Bacillus subtilis* and *Lactobacillus fermentum* with low IC₅₀ values from 2.58 ÷ 10.81 µg/ml.

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