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SYNTHESIS OF COMPLEX COMPOUNDS OF Cr(III) METAL IONS AND THEIR APPLICATION AS FLUOROSENSORS OF Pb(II) AND Cu(II) IN LABORATORY WASTE

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Abstract

Indonesia's exposure of lead (Pb) and copper (Cu) heavy metal ions to humans and the environment has received increased attention over the past few decades as a result of the increasing application of heavy metals in various industries and transportation. Complex compounds of Cr^{3+} sensors with fluorescence-based pyrazoline-derived ligands (fluorosensors) have the potential to detect Pb^{2+} and Cu^{2+} heavy metal ions. The purpose of this study is to synthesize complex compounds of Cr^{3+} ions with pyrazoline-derived ligands and its potential as a fluorescence-based sensor of Pb^{2+} and Cu^{2+} heavy metal ions. Complex compounds formed are characterized and fluorosensor study by spectroscopic methods. The results showed that the synthesis of complex compounds was successfully carried out and a brown precipitate with a melting point of 253.2°C was obtained. Based on the results of the characterization of complex compounds of Cr^{3+} metal ions with pyrazolin-derived ligands using a UV-Vis spectrophotometer, two absorption peaks were obtained at 226 and 370 nm, two peaks indicating the presence of transition types $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$. The fluorescence spectrophotometer results showed two emission peaks, the peak absorption maximum of Cr^{3+} complex compounds was in the region 491 nm with a fluorescence intensity of 320.40 a.u. The determination of the fluorosensor potential of Cr^{3+} complex compounds with the addition of Pb^{2+} and Cu^{2+} metal ions showed significant changes in fluorescence so that fluorosensor types were obtained for Cr^{3+} metal ion complex compounds with Pb^{2+} and Cu^{2+}, namely "turn-on" and "turn-off", respectively.

INTRODUCTION

Indonesia's exposure to lead (Pb) and copper (Cu) heavy metal ions to humans and the environment has received increased attention over the past few decades as a result of the increasing application of heavy metals in various industries and transportation [1]. The toxicity of heavy metals copper (Cu) and lead (Pb) in high doses can have several consequences in the human body that can affect central nervous function causing mental disorders, damaging blood constituents and can damage the lungs, liver, kidneys, and other vital organs, while in the environment it can cause pollution and bioaccumulation in living organisms [2].

Fluorescence-based sensors have been widely used in various fields, such as biochemistry, biomedicine, and clinical studies, as well as environmental pollution issues, such as the detection of Pb^{2+} and Cu^{2+} heavy metal ions [1]. This is because fluorescence-based sensor applications have the advantages of high sensitivity and selectivity, low cost and fast detection [3]. The complex compounds used can be used as fluoresensors if the compounds are aromatic, heterocyclic, or conjugated molecules. Complex compounds can be formed because there are metals and ligands that form coordinated covalent bonds [4]. Pyrazoline-derived ligan is a five-membered heterocyclic ring compound and contains two nitrogen atoms that act as free electron donors that can coordinate and chelate the central metal ion, namely Cr^{3+}, thus causing the formation of light absorption and emission and strong fluorescence properties because both have fluorescence properties. In addition, because chromium metal ions have a high affinity for pyrazoline-derived ligands as N-donors, ligands with pyrazolin rings can affect the photo-physical properties of chromium metal ions [4,5].

According to research by Zulys and Rachmawati (2017), the Eu^{3+} complex synthesized for the Pb^{2+} and Cd^{2+} ion fluorisensors showed that the addition of Pb^{2+} and Cd^{2+} metal ions acted as fluorosensors with turn-on and turn-off types, respectively [6]. Syahputri & Kusumawardani

(2021) successfully synthesized a pyrazoline-derived ligand, namely para-di-2-(1-phenyl-3-pyridil-4,5-dihydro-1H-pyrozol-5-il)benzene and complexed with cobalt metal resulting in a fluorescence intensity of 1150 a.u at a wavelength of 470 nm [7]. To the author's knowledge, there has been no research on the complex compound of Cr^{3+} metal ions with the para-di-2-(1-phenyl-3-pyridyl-4,5-dihydro-1H-pyrozol-5-il)benzene ligand as a fluorosensor for heavy metal ions Pb^{2+} and Cu^{2+}. Based on the background that has been described, this study aims to complex pyrazoline-derived ligands, namely para-di-2-(1-phenyl-3-pyridyl-4,5-dihydro-1H-pirazol-5-il)benzene with Cr metal and its potential as a fluorosensor for heavy metal ions Pb^{2+} and Cu^{2+}. The use of Cr metal as the central atom in complex compounds is expected to increase the fluorescence intensity so that it has the potential to be a fluorescence sensor to detect heavy metal ions Pb^{2+} and Cu^{2+}.

EXPERIMENT

The research began with the synthesis of pyrazoline-derived ligands using the synthesis method in the previous study in reference no. [7,8] After that it was continued with the synthesis of complex compounds using the method in reference no. [9,10], then continued with the application of complex compounds into fluorosensors of Pb^{2+} and Cu^{2+} heavy metal ions.

Material

The material used are methanol (pa Merck), chloroform (pa Merck), $Cr(NO_3)_3.9H_2O$ (pa Merck), $Pb(NO_3)_2$ (pa Merck), n-hexane (pa Merck), and $Cu(NO_3)_2$ (pa Merck).

Instrumentation

The instruments used are Melting Point Apparatus (RY-2), Fourier Transform Infra Red (Shimadzu IR Spirit), UV-Vis spectrophotometer (Shimadzu UV-1800), and fluorescence spectrophotometer (Hitachi F-2700).

Procedure

The synthesis of a complex compound from metal $Cr(NO_3)_3.9H_2O$ with the ligand para-di-2-(1-phenyl-3-pyridyl-4,5-dihydro-1H-pyrazole-5-yl)benzene and characterized using FTIR, UV-Vis spectrophotometer, and spectrofluorometer. The results of the synthesis of complex compounds were analyzed for their potential as fluorosensors against heavy metal ions Pb^{2+} and Cu^{2+} using a spectrofluorometer.

Synthesis of Cr^{3+} Complex Compound

A total of 1 mmol of the ligand and 1 mmol of $Cr(NO_3)_3.9H_2O$ were dissolved in 20 ml of methanol and chloroform with a 1:1 ratio in a beaker glass and stirred using a magnetic stirrer for 35 minutes at room temperature and pressure. After the precipitate was obtained, it was filtered and washed using cold methanol and cold hexane and then allowed to stand in a desiccator, then a metal:ligand stoichiometry analysis was carried out using the Job method according to the research in reference no. [11].

Compound Characteristics

The resulting complexes were characterized by melting point apparatus, foutier transform infrared spectrophotometer (FTIR), UV-Vis spectrophotometer, and fluorescence spectrophotometer. Furthermore, a fluorosensor study of complex compounds was carried out with the addition of heavy metal ions Pb^{2+} and Cu^{2+} using a fluorescence spectrophotometer, which started by dissolving complex compounds in a mixed solvent of methanol and chloroform (1:1, v/v) at a concentration of complex compounds of 10^{-5} M, then scanning at a wavelength of 220-650 nm. Next, complex compounds at a concentration of 10^{-5} M are dissolved in chloroform and methanol (1:1, v/v) then Pb^{2+} metal ions are added to it in the concentration range of 10^{-7}-10^{-4} M. The same is applied to Cu^{2+} metal ions. The scan was performed at a wavelength of 220-650 nm with a fluorescence spectrophotometer.

RESULT AND DISCUSSION

Synthesis of Complex Compound

The results of the synthesis of complex compounds of Cr^{3+} metal ions with pyrazoline-derived ligands were obtained in the form of brownish-orange solids with a melting point of 268.8ºC for complex compounds, while the melting point of ligands was 230.9ºC. The melting point of complex compounds is greater than the melting point of ligands, indicating that complex compounds have been formed. The results of stoichiometry analysis using the Job method can be seen in **Figure 1**.

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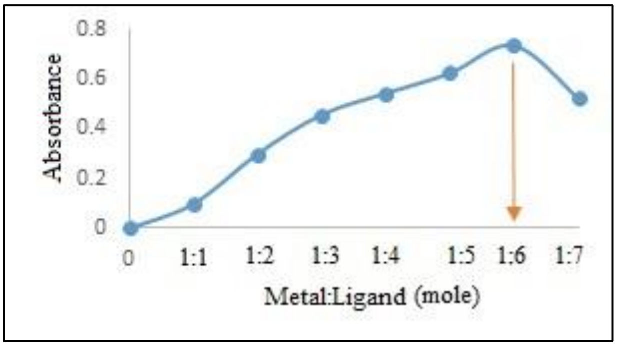

Figure 1. Job method graph.

In **Figure 1**, it shows that in a ratio of 1:6. is an appropriate comparison for the formation of complex compounds of Cr with pyrazoline-derived ligands, where 1 mole of Cr metal can bind 6 moles of ligands. In a ratio of 1:7, there is a decrease in absorbance due to the interaction between the metal d-d orbital energy and the π of the ligand which results in the transfer of electrons from metal to ligand, so that there is a change in absorbance or commonly called intensity reduction (quenching). Cr^{3+} complex compounds with a coordination number of 6 can be estimated to have an octahedral geometric structure [12].

Complex Compound Analysis and Application

The results of the analysis of complex compounds using the FTIR instrument can be seen in **Figure 2**.

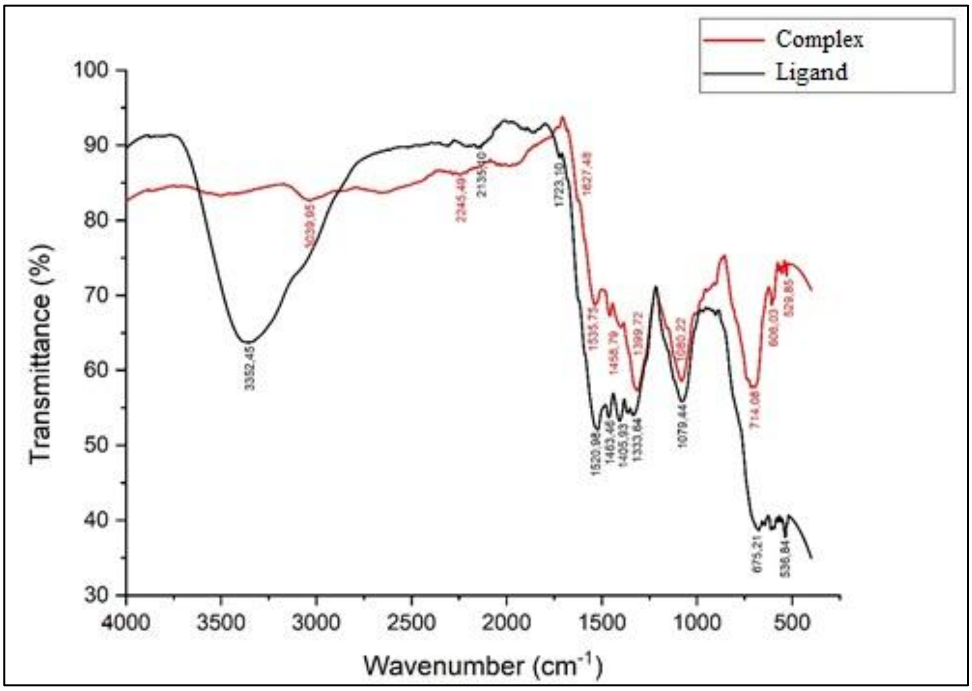

Figure 2. FTIR spectrum of complex compound and ligand.

Based on the IR spectrum of complex compounds in **Figure 2**, a wavenumber of 3039.95 cm^{-1} is obtained which indicates the presence of pyridine aromatic C-H absorption bands and at wavenumber 2245.49 cm^{-1} is an aromatic C=C bond [13]. The wavenumber 1627.48 cm^{-1} is the C=N bond [14]. The wavenumber of 1080.22 cm^{-1} indicates the presence of stretching vibration a C-N bond [15], while the wavenumber of 714.08-606.03 cm^{-1} indicates a C-H bond of Aromatis out-of-plane-bending [16]. The formation of complex compounds is characterized by the presence of a bond between Cr-N (N atoms derived from ligans) at a wave number of 529.85-339.44 cm^{-1}. In pyrazoline-derived ligands, the presence of conjugated electrons in the aromatic ring system of pyrazoline and pyridine ligands also affects the strength and stability of the ligands in forming a complex compound. This happens because the aromatic ring on the ligand has a bond whose electrons are delocalized. The delocalized electrons in the aromatic ring will increase the bond strength that occurs in the ligand and the central atom so that the more electrons are delocalized, the stronger the bond between the ligands and the central atom [17].

The formation of complex compounds can be initiated by wavelength scanning, then maximum wavelength measurements in the range of 190-550 nm are carried out for complex compounds of Cr^{3+} metal ions and para-di-2-(1-phenyl-3-pyridyl-4,5-dihydro-1H-pyrozol-5-il)benzene ligands shown in **Figure 3**.

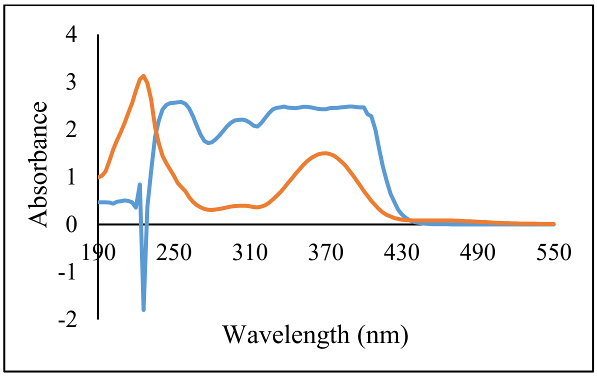

Figure 3. UV-Vis spectrum of ligand (blue) and complex compound (orange).

Based on **Figure 3**, there are three maximum wavelength peaks for ligands in the 223 nm, 256 nm, and 304 nm regions were obtained with a yellow solution. At the maximum wavelengths of 223 nm and 256 nm, the absorption process of the absorption band of the aromatic chromophore group, namely the benzene group, occurs, and at the wavelength of 304 nm the absorption process of the absorption band of the chromophore group of the ligand, namely the pyridine group and the pyrazoline group, the absorption band of the pyridine and pyridine chromophore group in the pyrazoline and pyridine derivative ligands is in the range of the maximum wavelength range of 300-400 nm [18].

Table 1. The UV-Vis absorption peak of complex compound.

Absorbance	λ max, nm (ε, M^{-} cm^{-})
3.084	226 (61680)
1.877	370 (37540)

In the spectrum of the complex compound Cr^{3+} produces two significant absorption peaks, where at the region of 226 nm it is a transition of π-π* (C=C), and in the region of 370 nm it is a transition n-π* containing free electrons on the donor atom N. The maximum wavelength of the transition π-π* is lower than that of the transition n-π* because for an electron to experience excitation from the π orbital to the π* orbital requires more energy than the excitation of electrons from the n (non-bonded) orbital towards the orbital π* [19].

Fluorescence analysis of complex compounds using a fluorescence spectrophotometer is used to look at the fluorescence intensity of complex compounds. In this study, observations have been made on the fluorescence characteristics of Cr^{3+} complex compounds with pyrazolin-derived ligands, which can be seen in **Figure 4**.

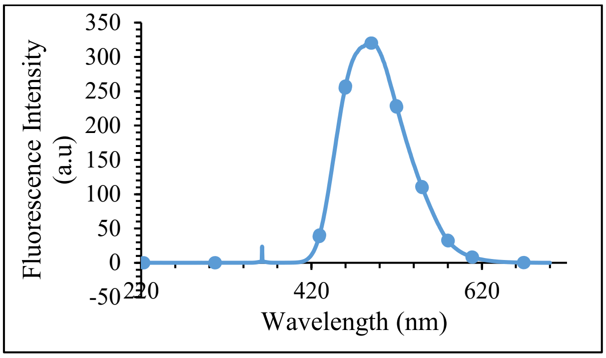

Figure 4. Fluorescence spectra of complex compound.

The results obtained based on **Figure 4** showed that two peaks of complex compound emissions were formed, namely in the 360 nm region with a fluorescence intensity of 23 a.u and in the 491 nm region with a fluorescence intensity of 320 a.u. The relatively large molar absorptivity (**Table 1**) indicates that the complex compounds formed have a strong fluorescence intensity [19].

The results of the fluorescence intensity of Cr^{3+} complex compounds with the addition of Pb^{2+} and Cu^{2+} ions can be seen in **Figures 5** and **6**. The concentration range of added heavy metal ions selected based on previous research by Syahputri & Linda (2021) [11].

The addition of Pb^{2+} heavy metal ions in the concentration range of $5x10^{-7}$-$5x10^{-4}$ M (**Figure 5**), experienced an increase in fluorescence intensity (turn-on), i.e. the illumination effect increased with the increase in the concentration of Pb^{2+} metal ions. The magnetism of a metal affects its fluorescence intensity, Pb^{2+} metal ions are diamagnetic metals that are more likely to increase fluorescence intensity [1]. Diamagnetic compounds do not have paired electrons, which means they cannot facilitate intersystem crossing and non-radiative decay that can reduce fluorescence. Therefore, in the absence of the intersystem crossing mechanism, the lifetime will be longer, resulting in an increased chance of fluorescence. Additionally, diamagnetic compounds can weakly interact with fluorescent molecules (for example, through coordination) and help maintain the electronic structure stable in an excited state [20]. On the other hand, the addition of Cu^{2+} heavy metal ions in the concentration range of 10^{-7}-10^{-4} M (**Figure 6**) decreases the fluorescence intensity (turn-off) along with the increase in the concentration of Cu^{2+} metal ions. This is also related to the magnetic properties of Cu^{2+} metal ions, namely paramagnetic, where paramagnetic metals are more likely to extinguish fluorescence intensity [21].

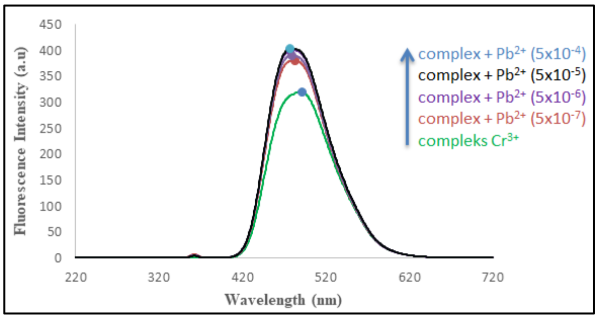

Figure 5. Fluorescence intensity spectrum of complex on the addition of heavy metal ions Pb^{2+} at concentrations $5x10^{-7}$-$5x10^{-4}$ M.

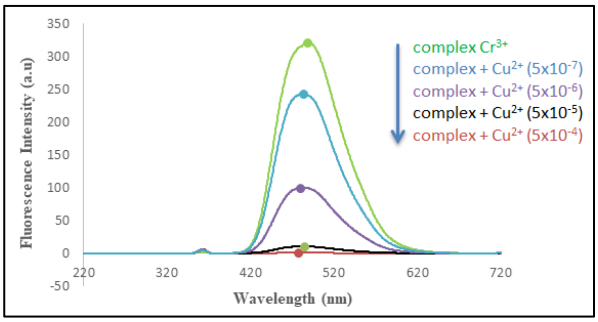

Figure 6. Fluorescence intensity spectrum of complex on the addition of heavy metal ions Cu^{2+} at concentrations $5x10^{-7}$-$5x10^{-4}$ M.

The increase and decrease in fluorescence intensity upon the addition of heavy metal ions Pb^{2+} and Cu^{2+} can be seen in **Table 2**, where the intensity of the complex compound before the addition of heavy metal ions is 320 a.u.

Table 2. Summarizing fluorescence intensities after Pb^{2+} and Cu^{2+} addition.

Concentration of Metal Ions (M)		Fluorescence Intensity (a.u.)
Pb^{2+}	$5x10^{-7}$	380
	$5x10^{-6}$	390
	$5x10^{-5}$	400
	$5x10^{-4}$	403
Cu^{2+}	$5x10^{-7}$	242
	$5x10^{-6}$	99
	$5x10^{-5}$	20
	$5x10^{-4}$	0.556

The selectivity of fluorosensors based on complex compounds occurs because certain metal ions can significantly decrease or increase the intensity of organic fluorophores. The addition of Cu^{2+} ions resulted in a quenching effect on the fluorescence intensity (turn-off), as Cu^{2+} is a quenching agent that directly contacts the chromium complex compound, leading to a decrease in fluorescence intensity. The quenching agent in the form of heavy metals or paramagnetic elements such as Cu^{2+} is known to cause a quenching of fluorescence intensity through the phenomenon of intersystem crossing [20]. Because the emission rate produced is low, the de-excitation process from the triplet state can be easily quenched by Cu^{2+} ions, at concentrations of 5×10^{-4} to 5×10^{-7} M experiencing a significant decrease in intensity, hence the addition of Cu^{2+} metal to the complex compound can be said to be selective. The sensitivity of fluorosensor to chromium complex compounds with the addition of heavy metal ions Pb^{2+} and Cu^{2+} can also be influenced by concentration; this is caused by the addition of Pb^{2+} ions at a concentration of 5×10^{-7} M (the lowest concentration) which can provide a response in the form of high fluorescence emission intensity. Thus, the addition of Pb^{2+} ions is more sensitive compared to the addition of Cu^{2+} ions at the same concentration (5×10^{-7} M). The determination of LOD, LOQ, emission lifetime, and selectivity towards the presence of other metal ions such as Zn^{2+} and Ni^{2+} in this study has not been observed and will be examined in future research.

CONCLUSION

The synthesis of complex compounds was successfully carried out and a brownish-orange precipitate with a melting point of 268.8°C was obtained. The formation of complex compounds is characterized by the presence of Cr-N bonds at wave numbers 529.85-339.44 cm^{-1}. The results of UV-Vis spectroscopy studies with a concentration of 10^{-5} M obtained two absorption peaks at 226 nm and 370 nm. The results of the complex compound fluorescence spectrophotometer study obtained a fluorescence intensity of 320 a.u. at a wavelength of 491 nm. The results of the fluorosensor complex compound study show that the addition of Pb^{2+} heavy metal ions can be used as a turn-on type fluorosensor, while complex compounds with the addition of Cu^{2+} heavy metal ions can be used as a turn-off type fluorosensor.

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