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# Synthesis and Characterization of Schiff Base from 4,4-Diaminodiphenyl Ether and Vanillin and Its Interaction with $Cu^{2+}$ Metal Ion

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## Abstract

The Schiff base has synthesized from the reaction between 4,4-diaminodiphenyl ether and vanillin. The resulting Schiff base acts as a ligand and interacts with $Cu^{2+}$ metal ions to form a complex compound. The Schiff base was analyzed using UV-visible and FT-IR spectroscopy, as well as X-ray diffraction (XRD). The stability of the Schiff base under different pH conditions was investigated, along with the interference effects of $Cd^{2+}$ and $Zn^{2+}$ ions on the formation of Schiff base complexes with $Cu^{2+}$. The formation of a yellow solid crystal indicated the successful synthesis of the Schiff base. The appearance of maximum absorption at 250 nm on the UV-Vis spectra signifies the electronic transition from $\pi$ to $\pi^*$. Absorption spectra at 1600 $cm^{-1}$ indicate the presence of an azomethine group. The diffraction pattern showed a sharp peak at an angle of $2\theta$= 19.301°, 51.04° for the Schiff base 4,4-diamino diphenyl ether-vanillin. Schiff base compounds exhibit the highest stability at pH 5, where the C=N double bond formed is more stable than other pH. Schiff base ligands form complexes with $Cu^{2+}$ metal ions, characterized by absorption in the charge transfer region (LMCT) at $\lambda$ 400 nm and the d-d transition at $\lambda$ 630 nm. The presence of $Cd^{2+}$ and $Zn^{2+}$ metal ions shifting absorption of the Schiff Base-$Cu^{2+}$ ligand complex towards shorter wavelengths (hypsochromic effect).

*Keywords: Schiff base, 4,4-diamino diphenyl ether, vanillin, $Cu^{2+}$ ion complex, $Cd^{2+}$ and $Zn^{2+}$ interference.*

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## Abstrak (Indonesian)

Telah dilakukan sintesis basa Schiff melalui reaksi antara 4,4-diaminodifenil eter dan vanilin. Hasil sintesis ini kemudian dipelajari untuk melihat interaksinya dengan ion logam $Cu^{2+}$. Karakterisasi basa Schiff dilakukan menggunakan spektrofotometer UV-Visible, FT-IR, XRD, serta pengaruh variasi pH. Interaksi antara basa Schiff dan ion logam $Cu^{2+}$ diteliti melalui pengaruh gangguan dari ion logam $Cd^{2+}$ dan $Zn^{2+}$ terhadap pembentukan kompleks basa Schiff dengan ion $Cu^{2+}$. Basa Schiff dari 4,4-diaminodifenil eter dan vanilin ditandai dengan terbentuknya kristal padat berwarna kuning. Adanya serapan maksimum pada panjang gelombang 250 nm, yang menunjukkan transisi elektronik dari $\pi$-$\pi^*$. Selain itu, spektrum FT-IR menunjukkan puncak serapan pada 1600 $cm^{-1}$, yang menandakan keberadaan gugus azometin (-C=N). Pola difraksi basa Schiff 4,4-diaminodifenil eter-vanilin memperlihatkan puncak pada sudut $2\theta$ = 19,301° dan 51,04°. Basa Schiff ini memiliki serapan tertinggi pada pH 5, menunjukkan bahwa ikatan rangkap (C=N) yang terbentuk lebih stabil dan memiliki ketahanan struktur yang baik. Ligan basa Schiff membentuk kompleks dengan ion logam $Cu^{2+}$, yang ditandai dengan munculnya serapan di daerah transfer muatan (LMCT) pada $\lambda$ = 400 nm dan transisi d-d pada $\lambda$ = 630 nm. Adanya ion logam $Cd^{2+}$ dan $Zn^{2+}$ menyebabkan pergeseran panjang gelombang serapan kompleks ligan basa Schiff-$Cu^{2+}$ ke arah panjang gelombang yang lebih pendek (efek hipsokromik).

*Kata Kunci: basa Schiff, 4,4-diaminodifenil eter, vanilin, kompleks ion $Cu^{2+}$, interferensi $Cd^{2+}$ dan $Zn^{2+}$.*

## INTRODUCTION

In 1864, German chemist Hugo Schiff introduced a new class of organic compounds called imines, later known as Schiff bases. These compounds are characterized by the (-CH=N) bond, formed by the condensation reaction between aldehydes and primary amines. Schiff bases play a crucial role in various fields, particularly coordination chemistry, where they act as ligands to form complexes with diverse structures involving transition metals [1]. Their ability to coordinate through the oxygen and nitrogen atoms in the azomethine group enables them to function as multidentate ligands, giving them flexibility, sensitivity, and selectivity toward metal atoms [2,3].

In the medical field, Schiff bases show promise as potential inhibitors for tuberculosis treatment and possess various biological activities, including antifungal, antitumor, antioxidant, antimalarial, antimicrobial, and antibacterial properties [4]. They are also valuable for detecting anions such as fluoride and cations like zinc, cadmium, and copper [5]. While copper is essential in small amounts, it becomes toxic at higher concentrations. Copper reacts with important elements and compounds like oxygen, sulfur, and nitrogen. Due to its reddish color, ductility, and malleability, excessive copper can lead to environmental pollution and pose risks to human health [6].

Vanillin *(3-methoxy- 4-hydroxybenzaldehyde)*, an organic compound with the molecular formula $C_8H_8O_3$, is the main component extracted from vanilla beans. It features three functional groups: aldehydes, ethers, and alcohols, with the reactive aldehyde group capable of forming C=N (imine) bonds with aromatic primary amines via addition-elimination reactions [7]. 4,4-Diaminodiphenyl ether, a diamine monomer with two primary amine groups ($-NH_2$), can be modified into its derivatives. The primary amines in the 4,4-diaminodiphenyl ether can react with the aldehyde group in vanillin to form Schiff base compounds [8]. The reaction between the $NH_2$ group of 4,4-diaminodiphenyl ether and the C=O group of an aldehyde forms a Schiff base. The aldehyde used in this reaction must be aromatic, as aromatic aldehydes offer a more stable conjugated structure than aliphatic aldehydes, which are less stable and prone to polymerization [9]. Vanillin is chosen because it is an aromatic aldehyde.

The Schiff base formed from 4,4-diaminodiphenyl ether and vanillin can serve as a chemosensor; the electron-donating and electron-accepting groups at both ends are connected by $\pi$-bond-containing groups that allow electron resonance during excitation [10]. Schiff base ligands are of significant interest in research due to their structural versatility and broad applications. Complexes formed with transition metal ions are highly effective in various chemical processes such as polymerization, oxidation, reduction, and catalysis.

Schiff base ligands possess unique chelating properties, allowing them to form bridging structures and offer multiple coordination modes, which enable the synthesis of homo- or heteronuclear complexes with central atoms. These ligands can donate more than one electron pair from their oxygen and nitrogen atoms to the d orbitals of transition metal ions, resulting in distinct structures and properties [11]. Many Schiff base ligands act as cation detectors for metal ions such as $Al^{3+}$, $Co^{2+}$, $Cu^{2+}$, $Cd^{2+}$, $Hg^{2+}$, $Ni^{2+}$, $Pb^{2+}$, and $Zn^{2+}$. They are also known for their ability to catalyse reactions and are widely used in biomimetic processes [6].

This research synthesis of the Schiff base compound (-N=CH-) was conducted between the aromatic aldehyde vanillin and the primary amine 4,4-diamino diphenyl ether. The resulting Schiff base was used as a detector for $Cu^{2+}$ metal ions. The Schiff base was characterized using a UV-visible spectrophotometer, Fourier transform infrared (FT-IR) spectroscopy, and X-ray diffraction (XRD). Additionally, the stability of the Schiff base structure under varying pH conditions was analysed, along with the interference effects of $Cd^{2+}$ and $Zn^{2+}$ions on the formation of Schiff base complexes with $Cu^{2+}$ using a UV-visible spectrophotometer.

## MATERIALS AND METHODS

### *Materials*

Copper (II) chloride ($CuCl_2.2H_2O$), hydrochloric acid (HCl), sodium hydroxide (NaOH), vanillin (Merck), 4,4-diamino diphenyl ether (Merck), distilled water, ethanol pa (96% Merck), dimethylformamide (DMF), and other supporting chemicals.

### *Synthesis of Schiff Base 4,4 diamino diphenyl ether - vanillin*

2 g (0.01 mol) of 4,4-diamino diphenyl ether, dissolved in 20 mL of distilled water, and 1.52 g (0.01 mol) of vanillin, dissolved in 20 mL of ethanol. Combine both solutions in a 250-mL Erlenmeyer flask with a condenser and magnetic stirrer. Heat the mixture under reflux at 80°C for 3 hours, then cool the mixture to room temperature. The solid product was filtered, washed with 10 mL of ethanol, and dried in an oven at 60 °C for 16 hours.

***Characterization of Schiff Base Using a UV-visible Spectrophotometer***

As much as 0.05 g of synthesized Schiff base was dissolved in 3 mL of DMF solvent. The solution was then measured using a UV-visible spectrophotometer in a range of 200–600 nm.

***Characterization of Schiff Base Using Fourier Transform InfraRed (FT-IR)***

The Schiff base was identified by using FT-IR Shimadzu Prestige-21. The synthesized Schiff base was mixed with KBr and then crushed in an agate mortar. The mixture was pressed and pelleted, then placed in the cell holder of the FT-IR instrument. The spectrum was measured in the wave number range 4000-400 $cm^{-1}$.

***Characterization of Schiff Base Using X-Ray Diffraction (XRD).***

The Schiff base sample is placed in the sample holder so that the X-ray radiation does not come out, the XRD must be tightly closed after the sample is placed. On the Personal Computer (PC) the angle of the goniometer or X-ray intensity detector is set, from this a graph of the relationship between the angle 2θ and the X-ray intensity will be obtained.

***The Effect of pH on the Structure of Schiff base***

Schiff base was dissolved using DMF solvent, whose pH was adjusted to pH 3, 5, 9, and 11 by adding 0.1 M HCl or 0.1 M NaOH. The absorbance of solutions was measured using a UV-visible spectrophotometer at a wavelength of 200-600 nm.

***The Effect of $Cd^{2+}$ and $Zn^{2+}$ metal ions Interference on the Formation of Schiff Base Complexes with $Cu^{2+}$***

For this experiment, solutions of $Cu^{2+}$, $Cd^{2+}$, and $Zn^{2+}$ metal ions were prepared in advance, each at a concentration of 10 mg/L. Next, 5 mL of the $Cu^{2+}$ solution was combined with 100 mg of Schiff base and analyzed using a UV-Vis spectrophotometer over a wavelength range of 200-800 nm.

To study the interference between metal ions, the following series of solutions were prepared:
a. 5 mL of $Cu^{2+}$ mixed with 5 mL of $Cd^{2+}$ solution
b. 5 mL of $Cu^{2+}$ mixed with 5 mL of $Zn^{2+}$ solution
c. 5 mL of $Cu^{2+}$ mixed with 5 mL each of $Cd^{2+}$ and $Zn^{2+}$ solutions.

The mixtures (a,b and c) were combined with 100 mg of Schiff base and analyzed using a UV-Vis spectrophotometer at a wavelength 200-800 nm.

***Analysis Data***

Analysis with UV-visible was used to determine electronic transitions that occur in Schiff bases and the effect of interference of $Cd^{2+}$ and $Zn^{2+}$ metal ions on the formation of Schiff base complexes with $Cu^{2+}$ metal ions.

## RESULTS AND DISCUSSION

***Synthesis of Schiff Base Compounds***

Schiff base is a compound synthesized from primary amines and aldehydes [1]. In this study, a Schiff base was synthesized from 4,4'-diamino diphenyl ether, a primary amine, and vanillin, an aldehyde compound. The crystal weight obtained was 3.1339 g and the yield percentage of 97.93%. Schiff base synthesis consists of two steps. The first stage is in the form of the addition of primary amines (as nucleophilic) to the -C=O carbon atom of vanillin and the second stage is in the form of an elimination stage which begins with the protonation of the -OH group, then it will be released as a water molecule which is a good leaving group can be seen in **Figure** 1 [4].

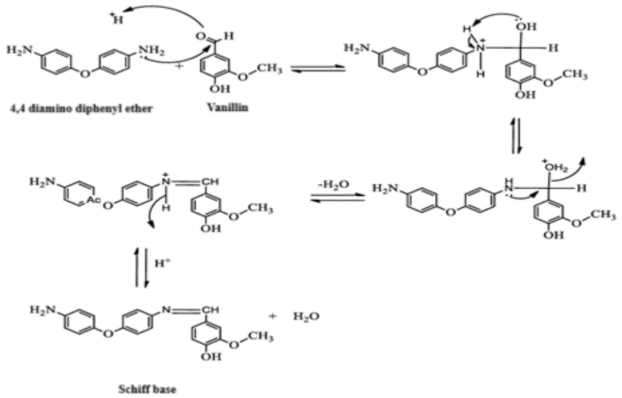


**Figure 1.** The Mechanism of Schiff Base Formation

***Characterization of Bases Schiff Using a UV-visible Spectrophotometer.***

This analysis was carried out at a wavelength of 200-400 nm. The resulting Schiff base spectrum is shown in **Figure** 2.

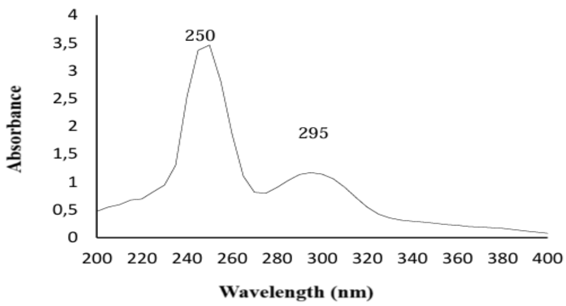


**Figure 2.** UV-Visible Spectrum of Schiff base in DMF Solvent

The imine group in the Schiff base undergoes an electronic transition $\pi \rightarrow \pi^*$. These characteristic peaks appear because the Schiff base has a chromophore group, namely azomethine, which absorbs UV light at that wavelength [12]. Based on the resulting spectrum, a maximum wavelength of 250 nm and 295 nm (π-π* transition) can be obtained.

***Characterization Results Using a Fourier Transform InfraRed (FT-IR) Spectrophotometer***

The Schiff base spectrum (**Figure** 3) shows the absorption of the azomethine functional group (-C=N) appears at wave number 1600 $cm^{-1}$. The azomethine group can be detected at wave numbers in the range of 1670-1560 $cm^{-1}$ [1,13]. The spectrum at wave number 3228 $cm^{-1}$ indicates the presence of OH vibration, and wave number 1622 $cm^{-1}$ indicates the presence of an aromatic C=C bond. The wave numbers between 1600-1400 $cm^{-1}$ contain the C=C functional group from rings of aromatic compounds [1]. In addition, in the spectrum of the Schiff base product, CO stretching vibrations also appear at wave number 1222 $cm^{-1}$. At wave number 3228 $cm^{-1,}$ it indicates an OH vibration, at wave number 1026 $cm^{-1}$ it indicates an O-CH $sp^3$ vibration. Wave number 3443 $cm^{-1}$ shows an overlap between $NH_2$.

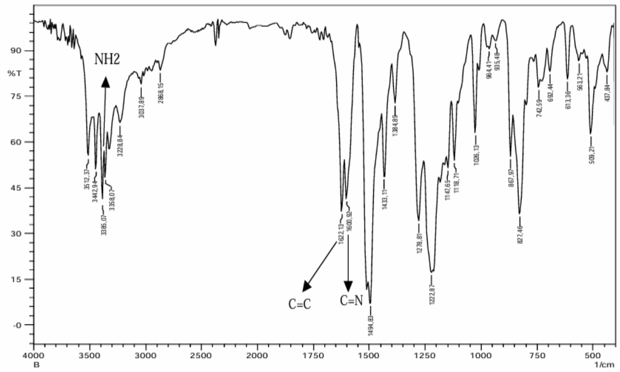


**Figure 3.** Schiff base Spectra

***Characterization Using X-Ray Diffraction (XRD)***

~~(~~XRD~~)~~ has a role as an analyzer of the composition of a phase or compound in the material characterization of crystals and identifying crystal changes in materials. Diffractogram of the Schiff base can be seen in **Figure** 4.

The XRD results obtained from the Schiff base 4,4-diaminodiphenyl ether-vanillin crystal powder have a sharp diffractogram peak at an angle of 2θ = 19.301°, 51.04°. Vanillin powder has a sharp diffractogram peak at 2θ = 13.85°, while 4,4 diamino diphenyl ether has a diffractogram peak of 2θ = 12.13°.

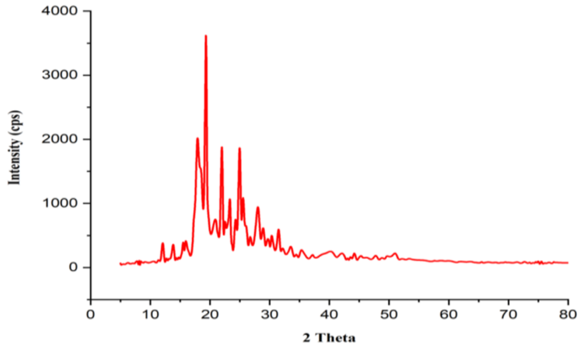


**Figure 4.** Schiff base diffraction pattern

***The Effect of pH on the Structure of Schiff Base Resistance***

The effect of pH on the Schiff base resistance structure is illustrated in **Figure** 5. From the data obtained, it can be concluded that the Schiff base synthesized from 4,4 diamino diphenyl ether and vanillin has the highest maximum absorption at pH 5. Imines at pH below 6.5 have structure imines with double bonds (C=N) that are more stable [13].

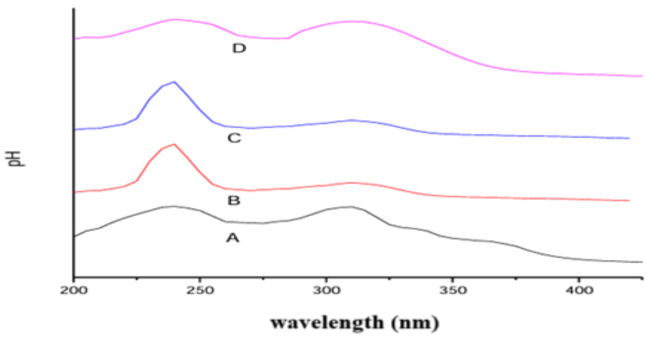


**Figure 5.** The Effect of pH on UV-visible spectrum of Schiff Bases at (a) pH 3; (b) pH 5; (c) pH 9; and (d) pH 11

The Schiff base compounds can act as ligands and chelators, so they can be used to detect metal ions including $Cu^{2+}$. Bonding between Schiff base compounds and $Cu^{2+}$ metal ions through coordination bonds. Donor atoms of Schiff base ligands are nitrogen atoms from the azomethine group and oxygen atoms from the methoxy vanillin group [14]. The color change occurs in the Schiff base from yellow to pale yellow after complexation with $Cu^{2+}$. The complex formation between the $Cu^{2+}$ metal ion and the Schiff base ligand can be seen in **Figure** 6.

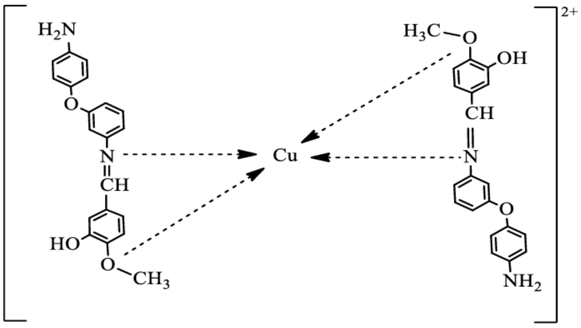


**Figure 6.** Formation Complex Compound between Schiff Base and $Cu^{2+}$ Metal Ions [14]

***Effect of Interference of $Cd^{2+}$ and $Zn^{2+}$ Metal Ions on the Formation of Schiff Base Complexes with $Cu^{2+}$***

The effect of interference of $Cd^{2+}$ and $Zn^{2+}$ metal ions on the formation of Schiff base complexes with $Cu^{2+}$ was carried out to determine the ability of Schiff base to react with $Cu^{2+}$ metal ions in the presence of other metals as interfering ions. This analysis was carried out by interacting Schiff base with a mixed solution of metal ions $Cu^{2+}$ and $Cd^{2+}$, $Cu^{2+}$ and $Zn^{2+}$, $Cu^{2+}$, $Cd^{2+}$, and $Zn^{2+}$ as well with the same concentration and volume as seen in **Figure** 7. $Zn^{2+}$ and $Cd^{2+}$ ions are ions that have potential as interfering ions in $Cu^{2+}$ analysis with Schiff Base.

Before interacting with $Cu^{2+}$ metal ion the Schiff base ligands had wavelengths at 250 nm and 295 nm respectively. After interacting with $Cu^{2+}$ metal ions (A), the Schiff base shifted its wavelength to 235 nm and 305 nm. The Schiff base ligand after being interacted with $Cu^{2+}$ metal ions also produce a new peak at 400 with a low intensity which is the electronic transition region for ligand to-metal charge transfer (LMCT). The d-d transition produced by the $Cu^{2+}$ metal ion as the central atom also appears at 630 nm an absorption from $^2Eg$ to $^2T2g$ transition that corresponds to octahedral geometry [15]. The appearance of the d-d transition in the 630 nm region indicates that a basic Schiff ligand $Cu^{2+}$ metal ion complex has been formed [16].

The Schiff base ligand complex with $Cu^{2+}$ metal ions also interacted with $Cd^{2+}$ and $Zn^{2+}$ metal ions to see the effect on the formation of the Schiff base ligand complex with $Cu^{2+}$ metal ions. After each interfering metal ion $Cd^{2+}$ (B), $Zn^{2+}$ (C), and $Cd^{2+}$ + $Zn^{2+}$ (D) were added to the Schiff base ligand complex-$Cu^{2+}$ metal ions produced the same wavelength in the Schiff base region, namely at 235 nm and 265 nm. Whereas in the charge transfer region (LMCT) the wavelengths formed in each complex are at 430 nm.

The wavelength of complex Schiff-$Cu^{2+}$ metal ions experience a shift towards a smaller wavelength after adding $Cd^{2+}$ and $Zn^{2+}$ metal ions called the hypochromic effect. The complex Schiff-$Cu^{2+}$ which has been added to $Cd^{2+}$ and $Zn^{2+}$ metal ions either added simultaneously or individually also did not produce a new peak in the d-d transition region at λ 630 nm which is the absorption of $Cu^{2+}$ metal ions. This indicates that the Schiff base ligands still form complexes with $Cu^{2+}$ metal ions in the presence of interference from $Cd^{2+}$ and $Zn^{2+}$ metal ions, at the charge transfer region (LMCT) λ 430 nm.

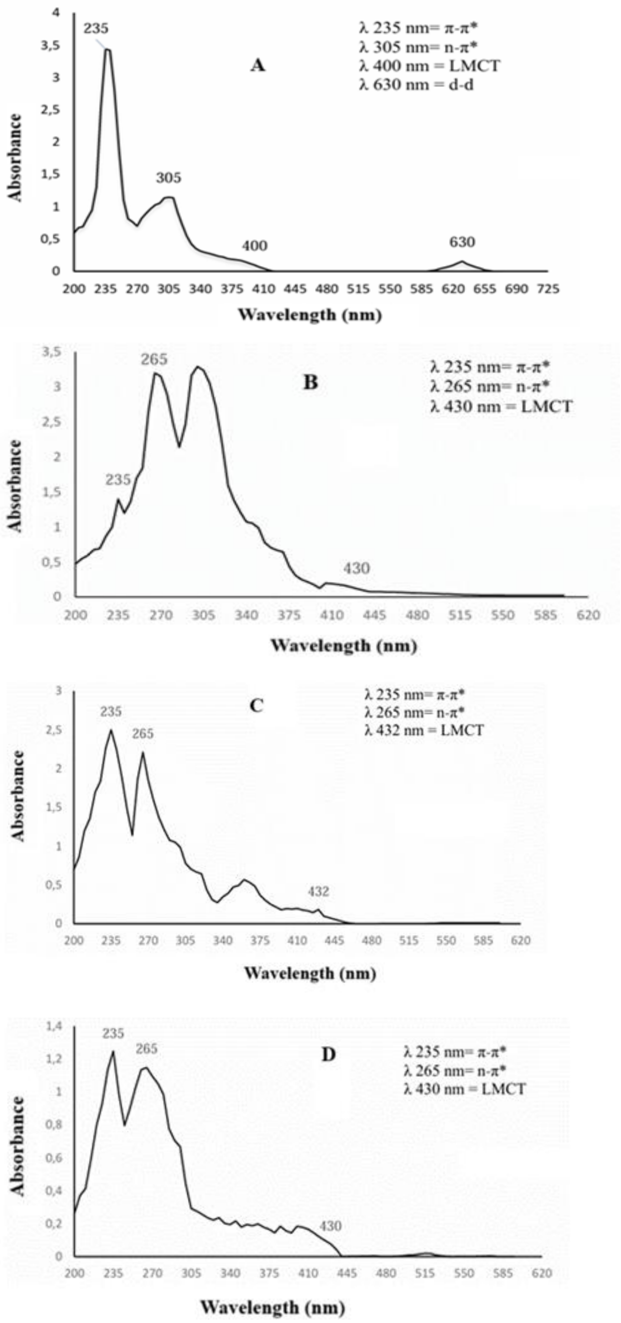


**Figure 7.** Effect of Interference of $Cd^{2+}$ and $Zn^{2+}$ Metal Ions on the Formation of Schiff Base Complexes with $Cu^{2+}$

## CONCLUSION

The Schiff base synthesis between 4,4-diaminodiphenyl ether and vanillin was successful. This is indicated by the formation of an azomethine group (C=N) at a wavelength of 1600 cm-1, characterization using a UV-visible spectrophotometer which shows an electronic transition $\pi \rightarrow \pi^*$ at λ 250 nm, whereas using XRD has a sharp diffractogram peak at an angle 2θ = 19.301°, 51.04°. 2. Schiff base compounds have the highest absorption at pH 5, where the double bond (C=N) that is formed is more stable or has a good resistance structure. 3. The Schiff base ligand-$Cu^{2+}$ metal ion after the complex interacted with interfering metal ions ($Cd^{2+}$ and $Zn^{2+}$) does not appear a dd transition region at λ 630 nm, which indicates that the Schiff base ligand still forms complexes with $Cu^{2+}$ metal ions in the presence of interference from $Cd^{2+}$ metal ions and $Zn^{2+}$, in the charge transfer region (LMCT) λ 430 nm.

## ACKNOWLEDGMENT

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