Influences of Ammonia for Synthesis of 8-Hydroxiquinoline Copper(II)

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Influences of Ammonia for Synthesis of 8-Hydroxiquinoline Copper(II)

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Abstract

Synthesis and characterization of complex compounds of 8-hydroxyquinoline copper (II) has been done. Synthesis of8- hydroxyquinoline copper (II) is done by mixing $CuSO_45H_2O$ with different concentration of ammonia and 8- hydroxyquinoline ligand in methanol. Product performed with magnetic stirrer, filtered, washed and dried in a desiccator. The results is obtained precipitated 8-hydroxyquinoline is copper (II) green-yellow. Characterization of complex compound was done by analysis of UV-Vis, AAS and FTIR. UV-Vis analysis results of 8-hydroxyquinoline copper (II) have a maximum wavelength at 394 nm, FTIR analysis results indicate the presence of N atom and O atom of C-O clusters in 8-hydroxyquinoline ligand was bonding with Cu (II)atom. If concentration of ammonia increasing, complex 8 – hydroxyquinoline copper (II) was obtaineddecreasing.

Keywords: Synthesis, complex compound Cu- 8-hydroxyquinoline

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Introduction

Oxine or 8- hydroxyquinoline (C_9H_7ON) forms sparingly soluble derivatives with metallic ions, which have the composition $M(C_9H_7ON)_2$ if co- coordination number of the metal is four (e.g., magnesium, zinc, cooper, cadmium, lead and indium), $M(C_9H_7ON)_3$ if the co- coordination number is six (e.g., aluminium, iron, bismuth, and gallium), and M $(C_9H_7ON)_4$ if the co-coordination number is eight (e.g., thorium and zirconium). There are, however, some exceptions, for examples, TiO $(C_9H_7ON)_2$, MnO_2 $(C_9H_7ON)_2$, WO_2 $(C_9H_7ON)_2$, and $UO_2(C_9H_7ON)_2$ (Vogel, 1978).

Copper (Cu)

Copper, silver and gold are in group 11 of the periodic table, and they share certain attributes. They have one s- orbital electron on top of filled d- electron shell and are characterized by high ductility and electrical conductivity. The filled d- shells in these elements do not contribute much to the inter atomic interaction, which are dominated by the s- electrons through metallic bonds. Unlike in metals with incomplete d-shells, metallic bonds copper are lacking a covalent character and are relatively weak [Huheey, 1981].

The softness of copper partly explains its high electrical conductivity (59.6 x 10^6 s/m) and thus also high thermal conductivity, which are the second highest among pure metals at room temperature. This is because the resistivity to electron transport in metals at room temperature mostly originates from scattering of electrons on thermal vibrations of the lattice, which relatively weak for a soft metal[Huheey, 1981].

Copper has 29 atomic number, they are 29 isotopes of copper. ^{63}Cu and ^{65}Cu are stable, with ^{63}Cu comprising approximately 69 % of naturally occurring copper. The other isotopes (^{62}Cu, ^{63}Cu and ^{67}Cu) are radioactive. Copper is present in the earth crust at a concentration of about 50 part per million (ppm), where occurs as native copper or in minerals such as the copper sulphides *chalcopyrite* or *chalcocite*, the copper carbonate *azurite* and *malachite*, and the copper (I) oxide mineral *cuprite* (Cotton and Wilkinson, 1988).

8- Hydroxyquinoline

The 8- hydroxyquinoline and its derivatives are widely used as analytical reagent [Raj, et all, 2001] and anti-amoebic agents, 8- hydroxyquinoline behaves as bidentate (N & O^-) univalent ligand to form chelates with several metals ions [Basollo, 1973]. 8-Hydroxyquinoline and its derivatives are widely used as ligand. 8- Hydroxyquinoline has 145,16 g/mol mass relatively, white powder, not soluble with aquadest, but soluble at organic solvent and acids, acetic acid for example (Underwood, 1980).

8- hydroxyquinoline behaves as bidentate (N & O^-) and used as ligand in synthesis complexes compounds. Structure of 8- hydroxyquinoline:

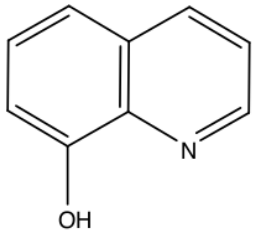

Figure 1. Structure if hydroxyquinoline

8-hydroxiquinoline has melting point 74-76 °C and has boiling point 276 °C, density 1.034 g/mol. The 8-hydroxiquinoline and this derivatives would be used as therapeutics as antibacterial, antifungal, as well as for treatment tuberculosis, diabetes and malaria (Soekardjo, 1995).

Derivatives 8-hydroxiquinoline have been used as tropical antiseptic and internal disinfectants that apparently exhibits low toxicity for human (Underwood, 1998).

This research Cu metals used as centre atom and 8-hydroxyquinoline used as ligand. 8- hydroxyquinoline ligand has O at CO bonding and N as electron donor [Huheey, 1981]. So bonding Cu metals and 8-hydroxyquinoline ligand has been obtained. "Continue Variation" method has done to form 8-hydroxyquinoline copper (II) (Sugiarto, 2009).

Synthesis 8- hydroxyquinoline copper (II) this research has done with mixing/ reaction $CuSO_4.5H_2O$ with 8-hydroxyquinoline (in methanol as solvent) in different ammonia concentration. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate grey-yellow was obtained, this is 8- hydroxyquinoline copper (II) complex.

Result 8- hydroxyquinoline copper (II) has analysed by UV-Vis, spectrophotometry, AAS and FTIR. from UV-Vis spectra wavelength maximum at 394 nm, FTIR analysed show interaction Cu metal with N atom and atom O at C-O bonding from 8-hydroxiquinoline is bonding as coordination bond. If concentration of ammonia increasing, complex 8 – hydroxyquinoline copper (II) was obtained decreasing.

Methodology

Materials

$CuSO_4.5H_2O$ p.a (Merck), Hydroxyquinoline p.a (Merck), methanol p.a (Merck), ammonia solution, aquabidest and aquadest.

Tool Volume pipet, beaker glass, analytical balance, magnetic stirrer, heating mantle, desiccator, funnel, reflux, FTIR spectrophotometer Shimadzu prestige 21, spectrophotometer UV-Vis Shimadzu 1601, atomic absorptions spectrophotometer (AAS), Whatmann paper.

Procedure

8-hydroxyquinoline Cuprum (II) synthesis

Becker glass I An aqueous solution contents of $CuSO_4.5H_2O$ (0.395 g) with ammonia concentration various (0,01M, 0,02M, 0,03M, 0,04M, 0,05 M and 0,07M) was soluble at 10 ml aquadest, Becker glass II Have contents 8- hydroxyquinoline soluble at methanol 10 ml. Becker glass I was added for Becker glass II dispensing drop by drop. Result obtained was reflux and mix by magnetic stirrer until 3 hour, and precipitate was obtained. Precipitated was filtered and wash by methanol and dried at desiccator until 3 days. Precipitate has grey- yellow colour, this is 8-hdroxyquinoline copper (II) complex. Base data, if concentration of ammonia increasing, Cu (II)- 8 – hydroxyquinoline complex was decreasing obtained, indicated with the colour grey-yellow for the complex is less. Analysis the complex was done with UV-Vis spectrophotometry, AAS, and FTIR.

Result and Discussion

Synthesis 8-hydroxyquinoline Copper (II) has done with mixing/ reaction $CuSO_4.5H_2O$ with 8-hydroxyquinoline (in methanol as solvent) in different ammonia concentration. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate grey-yellow was obtained, this is 8- hydroxyquinoline copper (II) complex.

Result 8- hydroxyquinoline copper (II) has analysed by UV-Vis, spectrophotometry, AAS and FTIR. from UV-Vis spectra wavelength maximum at 394 nm, FTIR analysed show interaction Cu metal with N atomic and atomic O at C-O bonding from 8-hydroxiquinoline as coordination bond.

Ammonia Variation

An aqueous $CuSO_4$ solution with ammonia concentration differently has result different. An aqueous $CuSO_4$ solution without ammonia the product is light blue. But if An aqueous $CuSO_4$ solution with ammonia the product is blue, this is tetramine copper (II) compound. If ammonia concentration is increase, the result 8-hydroxiquinoline copper (II) is decrease. This indicate by 8-hydroxiquinoline copper (II) grey-yellow is less. If ammonia concentration is increase, the system would be reaction Cu^{2+} with 8-hydroxiquinoline to form Tetramine Copper(II). So the 8-hydroxyquinoline copper (II) complex is decrease.

Ammonia Effect for 8-hydroxiquinoline copper (II) synthesis

From AAS data, If ammonia concentration is increasing, the result 8-hydroxiquinoline copper (II) is

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decreasing. This data would prove Cu^{2+} ion remainder obtained increasing, like table 1. This data would showed, if Ammonia concentration is increase, so Tetramine Copper(II) complex in solution is increase too. Copper(II) complex would be contribute to blocked Cu^{2+} ions to react with 8-hydroxiquinoline.

Table 1. Concentration Cu^{2+} at Variation Ammonia concentration

[Cu^{2+}] total	[Ammonia]	[Cu^{2+}] remainder
100 ppm	0.01 M	3.4 ppm
100 ppm	0.02 M	4.2 ppm
100 ppm	0.03 M	6.8 ppm
100 ppm	0.04 M	9.6 ppm
100 ppm	0.05 M	12.8 ppm
100 ppm	0.07 M	18.2 ppm

UV-Vis characterization

The adsorption and assignments related the ligand and the complex showed in Figure 2. Analysisspectrophotometry UV-Vis has used for establish λ_{max} (nm) sample. This wavelength used to trace λ_{max} (nm) $CuSO_4.5H_2O$ and λ_{max} (nm) 8-hydroxyquinoline copper (II) complex, after reaction was occurred.

The spectra where the electronic configuration of the metal d^{10}continuous adsorption of any (d -- d) transition. According the spectra data as well as those obtained from elemental analysis the chemical structure of the complex may be suggested for 8-hydroxyquinoline copper (II) complex. Change of wavelength, from 817 nm ($CuSO_4.5H_2O$) to 394 nm (8-hydroxyquinoline copper (II) complex) is indicated coordination bonding would be obtained. (Fessenden, 1986).

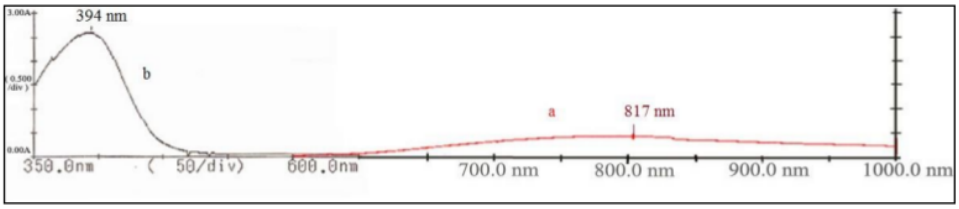

Figure 2 Spectra UV-VIS (a). $CuSO_4.5H_2O$, (b). 8-hydroksikuinolin copper (II) at methanol solvent

Transition electronic Energy 10Dq from $CuSO_4.5H_2O$ and 8-hydroxyiquinoline copper (II) showed table 3. Complex compounds usually has colour. Colour obtained not colour adsorption, but reflection from complement colour. Complement colour is overall wavelength from 8-hydroxiquinoline copper (II) complex. This complex have λ_{max} 394 nm, at UV area, because peak was obtained from ultraviolet UV-Vis area and adsorption wavelength from violet to blue (Fessenden, 1986).

Table 3:Wavelengthand 10Dq Energy for $CuSO_4.5H_2O$ and 8-hydroxyquinoline cuprum (II) complex

No.	Compound	λ_{max} (nm)	10Dq (KJ/mol)
1.	$CuSO_4.5H_2O$	817	146,288
2.	8-hydroxyquinoline copper (II) complex	394	303,343

IR characterization

The characteristic vibration and assignments of ligand 8-hydroyquinoline and complex compound are showed in figure 4 and analysed the IR spectra are described in table 5.

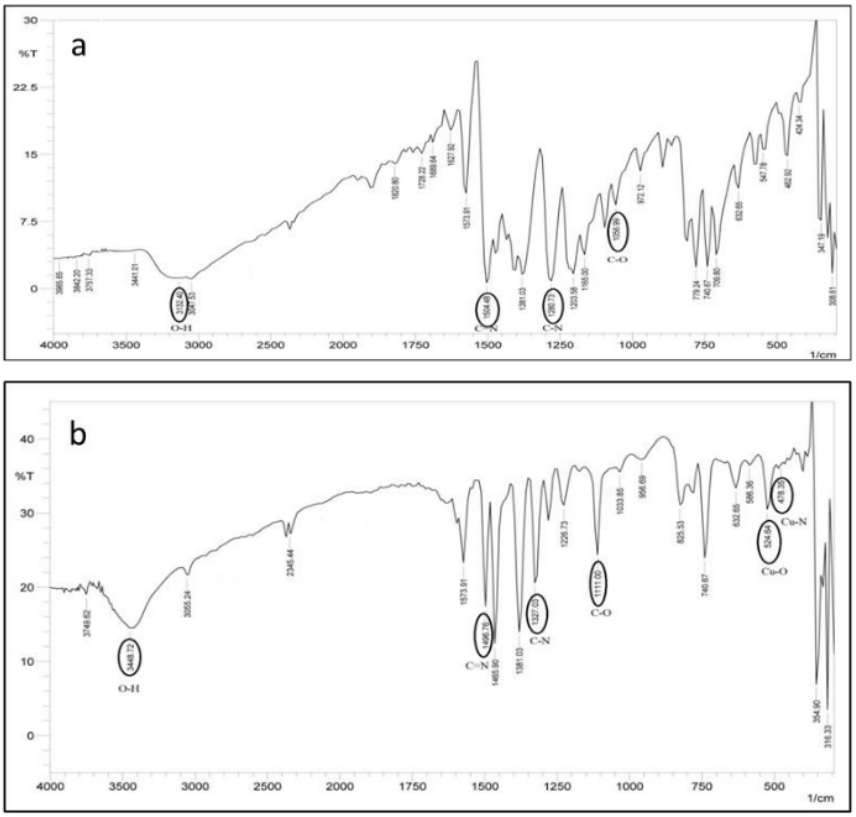

Figure 4 (a.) IR Spectra 8-hydroxyquinoline, (b.) 8 – hydroxyquinoline copper (II).

Table 5: IR spectra

Peak	8-hydroxyquinoline (cm^{-1})	8 -hydroxyquinoline copper (II). (cm^{-1})
v O-H stretch	3132.4	3448.7
v C=N stretch aromatic	1504.4	1496.7
v C-N stretch aromatic	1280.7	1327.0
v C-O stretch phenol	1056.9	1111.0
v Cu-N	-	478.3
v Cu-O	-	524.6

The spectrum of phenol exhibited the strong band of 1056,9 cm^{-1} for 8-hydroxy-quinoline and 1111 cm^{-1} for 8 -hydroxyquinoline copper (II). While another strong adsorption bands at 1280 cm^{-1} for 8-hydroxyquinoline and 1327 cm^{-1} for 8 -hydroxyquinoline copper (II).

The spectra of complex.

The spectra exhibited amarked difference the adsorption band (Figure: IV.3) belonging to be stretching vibration of V (C-O) of the carbonyl group have been found in the range between (1100 cm^{-1}-1200 cm^{-1}). According data, suggesting the possibility of the bonding complex compound is coordination bond of the ligand 8-hydroxyquinoline through the oxygen atomic in carbonyl group.

Adsorption assigned for V (C-N) was noticed at the range 1300 cm^{-1}-1350 cm^{-1} shifted to the higher frequencies. Adsorption assigned for V (Cu-N) was

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noticed at the range 478,3 cm^{-1} and V (C-O) was noticed at the range 500 cm^{-1} – 550 cm^{-1}, which indicate the coordination of nitrogen atomic of the V (C-N) group and oxygen atomic of the V (C-O) group to the central copper ion.

Conclusion

In this study,8- hydroxyquinoline (8HQ) copper (II) synthesis, were investigated:

1. $CuSO_4$ 5 H_2O has UV- Vis characteristic adsorption at λ_{max} 817 nm, and 8- hydroxyquinoline (8HQ) copper (II) has UV-Vis characteristic adsorbtion at λ_{max} 394 nm.
2. 8-hydroxyquinoline would coordinate bond with Cu^{2+} ion, by N atomic and O atomic.

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