

LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU PEER REVIEW
KARYA ILMIAH : PROSIDING

Judul Karya Ilmiah (Prosiding) : Influences of Ammonia for Synthesis of 8-Hydroxyquinoline Copper(II)
 Nama/ Jumlah Penulis : Suhartana, Laelatri Agustina, Sriatun (3)
 Status Pengusul : Penulis pendamping
 Identitas Prosiding : a. Judul Prosiding : Green Chemistry: Proceeding of 9th Joint Conference on Chemistry, 12-13 November 2014
 b. ISBN/ISSN : 978-602-285-049-6
 c. Thn Terbit, Tempat Pelaks. : 2015, Semarang
 d. Penerbit/Organiser : UNNES Press
 e. Alamat Repository/Web : https://jcc.undip.ac.id/24/the-proceeding-of-9th-jcc-semarang-2014.conf#post_detail
 Alamat Artikel : https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev_Part7.pdf
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<https://doc-pak.undip.ac.id/2874/29/Turnitin%20C29.pdf>
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 (beri ✓ pada kategori yang tepat) ☐ Prosiding Forum Ilmiah Nasional

Hasil Penilaian *Peer Review* :

Komponen Yang Dinilai	Nilai Maksimal Prosiding		Nilai Akhir Yang Diperoleh
	Internasional <input checked="" type="checkbox"/>	Nasional <input type="checkbox"/>	
a. Kelengkapan unsur isi prosiding (10%)	1,5		1,5
b. Ruang lingkup dan kedalaman pembahasan (30%)	4,5		4,3
c. Kecukupan dan kemutakhiran data/informasi dan metodologi (30%)	4,5		4,3
d. Kelengkapan unsur dan kualitas terbitan /prosiding (30%)	4,5		4,4
Total = (100%)	15,00		14,5
Penulis Pendamping: $(0,4 \times 14,5) / 2 = 2,9$			

Catatan Penilaian Paper oleh Reviewer :

1. Kesesuaian dan kelengkapan unsur isi prosiding:

Unsur isi prosiding sesuai dan lengkap. Nilai 1,5

2. Ruang lingkup dan kedalaman pembahasan:

Ruang lingkup tentang pengaruh ammonia pada pembentukan kompleks tetramin tembaga (II), yang mana kompleks ini digunakan untuk mensintesis 8-hidroksi kuinolin tembaga (II). Pembahasan cukup namun kurang didukung oleh Pustaka yang relevan. Nilai 4,3

3. Kecukupan dan kemutakhiran data/informasi dan metodologi:

Data dan informasi pada penelitian tersebut cukup, namun pustakanya kurang mutakhir, sedangkan metodolginya cukup memadai. Nilai 4,3

4. Kelengkapan unsur dan kualitas terbitan/ prosiding:

Unsur prosiding lengkap, kualitas prosiding baik. Nilai 4,4

Semarang,
 Reviewer 1



Dr. Bambang Cahyono, MS
 NIP. 196303161988101001
 Unit Kerja : Departemen Kimia FSM UNDIP

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	Internasional <input checked="" type="checkbox"/>	Nasional <input type="checkbox"/>	
e. Kelengkapan unsur isi prosiding (10%)	1,5		1,2
f. Ruang lingkup dan kedalaman pembahasan (30%)	4,5		4,0
g. Kecukupan dan kemutakhiran data/informasi dan metodologi (30%)	4,5		4,3
h. Kelengkapan unsur dan kualitas terbitan /prosiding (30%)	4,5		4,5
Total = (100%)	15,00		14,0
Penulis Pendamping: $(0,4 \times 14) / 2 = 2,8$			

Catatan Penilaian Paper oleh Reviewer :

- Kesesuaian dan kelengkapan unsur isi prosiding:**
 Unsur isi prosiding lengkap dan sesuai dengan turnitin similarity 18%. Nilai 1,2
- Ruang lingkup dan kedalaman pembahasan:**
 Ruang lingkup penelitian adalah sintesis tembaga 8-hidroksi kuinolin dan pengaruh ammonia pada riset tersebut. Kedalaman pembahasan kurang didukung oleh referensi terkait. Nilai 4,0
- Kecukupan dan kemutakhiran data/informasi dan metodologi:**
 Data yang disajikan cukup dan kemutakhiran kurang karena tidak didukung literatur terkini (kurang dari 10 tahun). Metodologi disajikan dengan runtut dan bisa diulang oleh peneliti lain. Nilai 4,3
- Kelengkapan unsur dan kualitas terbitan/ prosiding:**
 Kelengkapan unsur memadai dan kualitas terbitan cukup baik. Nilai 4,5

Semarang,
 Reviewer 2

Drs. Gunawan, M.Si, Ph.D
 NIP.196408251991031001
 Unit Kerja : Departemen Kimia FSM UNDIP

LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
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Hasil Penilaian *Peer Review* :

Komponen Yang Dinilai	Nilai Reviewer		Nilai Rata-rata
	Reviewer I	Reviewer II	
a. Kelengkapan unsur isi prosiding (10%)	1,5	1,2	1,35
b. Ruang lingkup dan kedalaman pembahasan (30%)	4,3	4,0	4,15
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)	4,3	4,3	4,3
d. Kelengkapan unsur dan kualitas terbitan/prosiding(30%)	4,4	4,5	4,45
Total = (100%)	14,5	14,0	14,25
Penulis Pendamping: $(0,4 \times 14,25) / 2 = 2,85$			

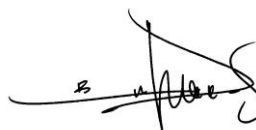
Semarang,

Reviewer 2



Drs. Gunawan, M.Si, Ph.D
 NIP. 196408251991031001
 Unit Kerja : Departemen Kimia FSM UNDIP

Reviewer 1



Dr. Bambang Cahyono, MS
 NIP. 196303161988101001
 Unit Kerja : Departemen Kimia FSM UNDIP

CERTIFICATE OF PRESENTATION

Certificate no.: 240/UN7.3.8/2014



The conference on *Green Chemistry*

This is to certify that

Suhartana

has presented a paper entitled

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

at the 9th Joint Conference on Chemistry held on 12-13 November
2014 in Semarang that organised by Chemistry Department,
Diponegoro University

Dean of Faculty of Science and Mathematics
Diponegoro University

Dr. Muhammad Nur, DEA
NIP 19371126 199001 1 001

Semarang, 13 November 2014

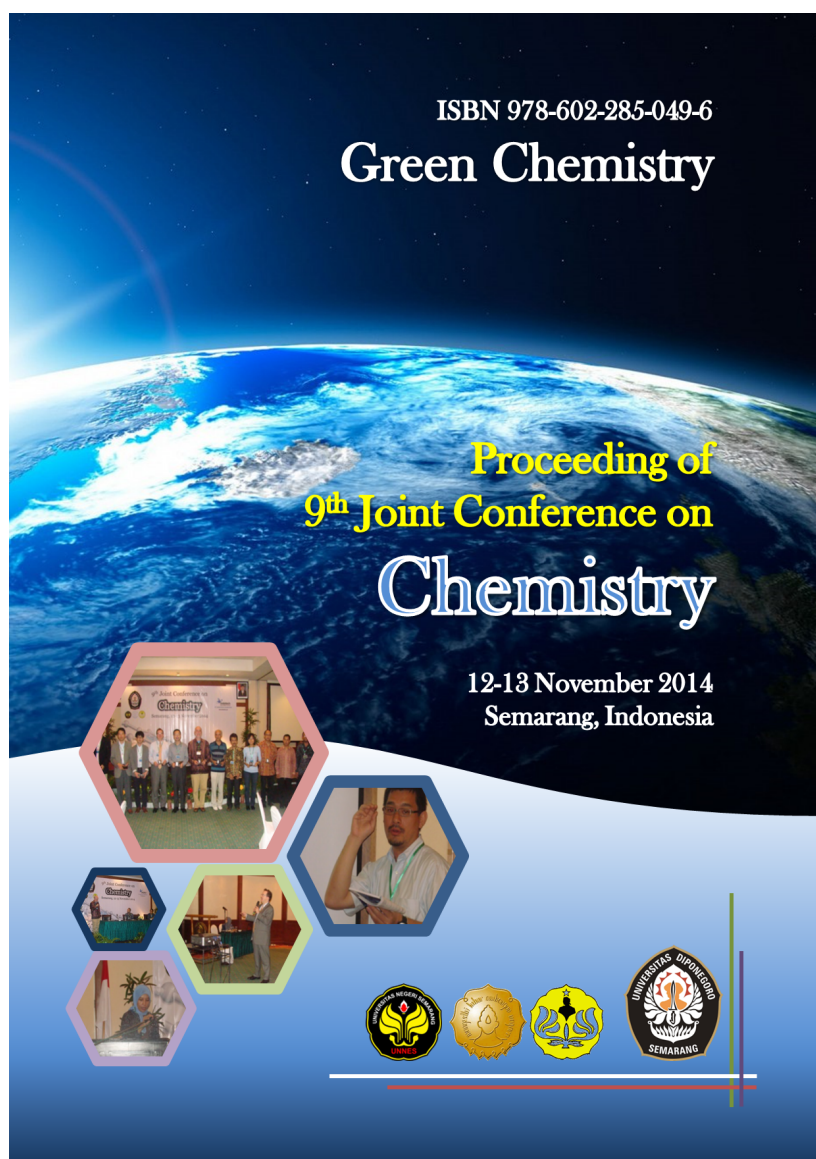
9th Joint Conference on
Chemistry Chair

Dr. Agustina L.N. Aminin, M.Si
NIP 19700801 199803 2 001



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Synthesis and Characterization of Nano Scale Zero-Valent Iron Supported on Mesoporous Silica

Atyaf Khalid Hammed^a, Nugroho Dewayanto^a, D. Dongyun^b, Mohd Ridzuan Nordin^a

Abstract

Nano scale zero-valent iron (NZVI) supported on mesoporous silica (MSN) was synthesized through liquid phase reduction route. In this method, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ solution was titrated with NaBH_4 as reduction agent. Various colours of products from black to light brown were formed from this process due to the different intensity of oxidation process. Mesoporous silica nanoparticles (MSNs) were prepared through the activation of fumed silica with concentrated hydrochloric acid (HCl). The mixture was refluxed at 90 °C with continuous stirring for 4 hours. This treatment increases the BET surface area from 61.0957 m^2/g to 125.8745 m^2/g . Preparation of NZVI supported by MSNs was conducted in alkaline solution. Amount of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in aqueous solution and activated silica in certain ratio was mixed at room temperature. NaBH_4 solution was added to the mixture in drop wise manner (3 ml/min) with vigorous stirring at room temperature. NZVI/MSN were characterized by XRD, BET, FTIR and FESEM. The capacity of NZVI/MSN in adsorption of methylene blue (MB) from aqueous solution was determined in series of batch experiments. Initial experiment showed the best performance of the adsorbent was achieved at $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ to MSN weight ratio of 0.4. The equilibrium was reached after 60 min of adsorption. The optimum adsorption condition was achieved at initial concentration 15 mg/L of MB and initial pH solution 7 under room temperature. NZVI/MSN is found to be an effective adsorbent for removing MB from aqueous solution.

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Introduction

The widespread application of dyes in textiles, printing, and food plants has produced a large amount of dye containing wastewater. Because some dyes and their degradation products may be carcinogens and toxic, the removal of dyes from wastewater becomes an important issue in environmental protection. Moreover, the colour that is generated by the presence of dyes in surface water causes great concern to the public. This urges an intensive search for the best available technology for the removal of dyes. Some physico-chemical methods, such as advanced oxidation and biological process, coagulants, oxidizing agents, membrane, electrochemical, and adsorption techniques have been proposed to satisfy the above requirements (Idris et al., 2007, Badruddoza et al., 2010, Kadirova et al., 2013). Among these methods, it was found that adsorption might be an efficient and economic process to remove dyes and also to control the biochemical oxygen demand (Ling et al., 2012).

Numerous studies have been done on dyes adsorption kinetics, equilibrium modelling, and mechanism as well

as to the factors that affect adsorption. Recently, mesoporous materials such as MCM-41 have also received a considerable recognition due to their large pore-space and special surface property (Petala et al., 2013). Porous materials have attracted the attention of scientists due to commercial interest related to their applications in separations, catalysts, and purification technologies. In the last decade, intensive scientific research efforts have been made in the areas of nanoporous materials (Zhu et al., 2009).

Nanostructures in the form of thin films, nanoparticles, nanocomposites and nanocrystalline materials are of interest for both fundamental scientific research and technological applications since some of their properties are controlled by their extremely large surface areas (Ray et al., 2010). Nanoporous and nanostructured materials are also considered ideal candidates for surface environment interactions, such as in gas-sensing, heterogeneous catalysis, and separation. Furthermore, scientists are still targeting new adsorbent with good property. In recent years, with the development of nanotechnology, various

Calcium Phosphate-Chitosan Composite as a Bone Cement CandidateTri Windarti^a and Benjamin Horrocks^b**Abstract**

A research about study of surface chemistry and morphology of bone cement from calcium phosphate-chitosan composite has been conducted. Surface chemistry and morphology of an artificial bone is directly influence to the osteoinductivity property. Calcium phosphate compounds as powder phase was reacted to liquid phase that contains of Na_2HPO_4 solution and chitosan. Analysis instruments such as XRD and SEM-EDS were used to identify surface chemistry, morphology and structural transformation of cement. Setting time and hardening time were tested to know the feasibility of cement when used in minimally invasive surgery technique. The results showed that setting time of calcium phosphate cement became shorter as well hardening time became longer by the increasing of liquid/powder (L/P) ratio and chitosan concentration. SEM image of calcium phosphate showed cuboid crystal shape. The transformation of calcium phosphate into CPC and composite were not changing its structure and morphology. The change only happen on calcium phosphate surface that caused by deposition of phosphate ions. It can be concluded that calcium phosphate – chitosan composite has osteoinductive property and can be used as bone cement.

Keywords: calcium phosphate, bone cement, composite

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Introduction

Calcium phosphate compounds (CP) are known as material that can accelerate recovery process of a damaged bone and formation of a new bone tissue (Ogose et al, 2006). The bioactivity and biocompatibility of CP are not only related to its chemical structure but also to the surface chemistry and morphology. Surface chemistry and morphology have strong effect to the ability of protein adsorption in vivo, cell adhesion and host response to implant (Ratner and Bryant, 2004). Generally, biomaterial for artificial bone is developed to have specific function and capacity to facilitate protein adsorption. Also, that biomaterial should be able to form composite with organic matrix such as chitosan, collagen or gelatine, because human bone is a composite of hydroxyapatite-collagen type I (Dee et al, 2002).

To improve the function of the CP as a biomaterial, calcium phosphate cement (CPC) has been developed. CPC are produced by chemical reaction of a solid phase (powder phase) and a liquid phase, which when mixed, form a paste which progressively hardens into a solid. The powder phase contain of one or several calcium phosphate compounds (CP) and the liquid phase is water or a calcium or phosphate containing solution. The dissolution of CP will produce oversaturation of

liquid phase, thus inducing the precipitation of crystals (Zang et al, 2014).

Beside the excellent biological behaviour, the other advantages of CPC is injectable and able to harden in vivo at body temperature. The viscous paste of CPC can be injected into damaged bone area and directly contact with the bone surface, so that CPC could be applied in minimally invasive surgery technique. This technique could reduce surgery duration, reduce the effect that caused by the opening of muscle tissue, reduce the wound and minimize pain (Ginebra et al, 2010). It is hoped that the patient will get a rapid recovery with lower costs.

In this research, chitosan was used as organic matrix to form calcium phosphate – chitosan composite. Chitosan is a biopolymer with chemical structure poly (2-amino-2-dioksi- β -D-Glucose) and has similarity with collagen structure. Chitosan has been widely used in medical field because of its properties such as biocompatible and biodegradable (Hargono et al, 2008). The addition of chitosan into CPC will affect to the surface structure and morphology of CPC that caused by interaction of powder phase-chitosan and liquid phase-chitosan.

Catalytic properties of bimetallic NiNP-M/AlOH (M = Sn, In, Ga, Ag, Nb, and Zr) on selective hydrogenation of furfural

Rodiansono^{a*}, M. D. Astuti^a, A. Ghofur^b, Shogo Shimazu^c

Abstract

A series of bimetallic NiNP-M/AlOH (M = Sn, In, Ga, Ag, Nb, and Zr) catalysts have been synthesised by a simple hydrothermal of the mixture of nickel nanoparticles supported aluminium hydroxide (NiNP/AlOH) and a salt solution containing those of the above metals at 423 K for 2 h. The synthesised catalysts were characterised by using ICP-AES, TG-DTA, XRD, N₂-adsorption, and H₂-chemisorption. The catalytic properties were evaluated on the selective hydrogenation of furfural (FFald) at 453 K for 90 minute. Reactant and products were characterised by GC, GC-MS, and ¹H NMR. The presence of the second metal remarkably reduced the crystallite sizes of Ni metal as indicated by the broadened diffraction peak of Ni(111) species compared to the former of NiNP/AlOH. H₂-uptake of Ni-M/AlOH also was lower than that of NiNP/AlOH. NiNP-Sn/AlOH and NiNP-In/AlOH catalysts showed extremely high selective hydrogenation towards furfuryl alcohol (FFalc) with almost 99% yield. We expect that the high activity and selectivity over Ni-Sn/AlOH and Ni-In/AlOH catalysts due to the formation of Ni-Sn or Ni-In alloys. On the other hand, NiNP-M/AlOH (M= Ga, Nb, Ag, Zr, and Ga) catalysts showed lower the catalytic activity than that of NiNP/AlOH catalyst. Therefore, further investigation of role of the second metal on the catalytic properties of NiNP-M/AlOH (M = Sn and In) catalysts is under progress.

Keywords: bimetallic catalysts, Ni-Sn alloy, Ni-In alloy, selective hydrogenation, furfural

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Introduction

Selective hydrogenation of furfural (FFald) to furfuryl alcohol (FFalc) is great industrial interest since it widely use in various applications[1]. Industrially, furfuryl alcohol was produced by liquid hydrogenation of furfural at the high temperature and H₂ pressure by using copper-chromite (Cu-Cr) catalysts which exhibits moderate in activity and selectivity. The main drawbacks of this catalyst system are toxicity and unrecyclable due to generated Cr₂O₃ and severe leaching of the metal into product[2-3]. Therefore, several attempts have been reported in order to replace Cu-Cr catalysts or to develop a new metallic catalyst system which have more efficient catalytic process and less severe of environmental problem.

Among developed metal catalysts, nickel-based catalyst with metal co-promotor or modified supports has been studied intensively due to its high activity for hydrogenation both of C=C and C=O. Several metal co-promoters were applied such as Cu [4, 8], Fe, Ce [5-7],

and Sn [10] in order to improve its chemoselectivity towards C=O rather than to C=C. In this advantage, system based on Ni modified with Fe, Ce or heteropolyacids have been proved to be successful, reaching 98% selectivity to FFA at almost total conversion [5-8]. However, in some cases these modified nickel catalysts cannot reuse [4] and also showed moderate in activity or selectivity [10]. Recently, Merlo et al. reported that tin modified of Pt/SiO₂ catalyst showed 96% selectivity to furfuryl alcohol and required 6 h to reach a complete reaction[9]. Moreover, the employing of noble metal catalyst is less favourable in economical advantageous. Therefore, the design less expensive the active and selective catalyst system for production furfuryl alcohol is an issue of interest, which still presents great challenges.

We recently have reported the chemoselective hydrogenation of FFald and various unsaturated carbonyl compounds over Ni-Sn catalysts both bulk and supported. The chemoselectivity of Ni-Sn alloy

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Introduction

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

Suhartana, Laelatri Agustina, Sriatun

Abstract

Synthesis and characterization of complex compounds of 8-hydroxyquinoline copper (II) has been done. Synthesis of 8-hydroxyquinoline copper (II) is done by mixing $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 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 obtained decreasing.

Keywords: Synthesis, complex compound Cu- 8-hydroxyquinoline

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Introduction

Oxine or 8-hydroxyquinoline ($\text{C}_9\text{H}_7\text{ON}$) forms sparingly soluble derivatives with metallic ions, which have the composition $\text{M}(\text{C}_9\text{H}_7\text{ON})_2$ if co-ordination number of the metal is four (e.g., magnesium, zinc, copper, cadmium, lead and indium), $\text{M}(\text{C}_9\text{H}_7\text{ON})_3$ if the co-ordination number is six (e.g., aluminium, iron, bismuth, and gallium), and $\text{M}(\text{C}_9\text{H}_7\text{ON})_4$ if the co-ordination number is eight (e.g., thorium and zirconium). There are, however, some exceptions, for examples, $\text{TiO}(\text{C}_9\text{H}_7\text{ON})_2$, $\text{MnO}_2(\text{C}_9\text{H}_7\text{ON})_2$, $\text{WO}_2(\text{C}_9\text{H}_7\text{ON})_2$, and $\text{UO}_2(\text{C}_9\text{H}_7\text{ON})_2$ (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 (59.6×10^6 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 , ^{64}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 al, 2001] and anti-moebic agents, 8-hydroxyquinoline behaves as bidentate ($\text{N} \& \text{O}^-$) 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 ($\text{N} \& \text{O}^-$) and used as ligand in synthesis complexes compounds. Structure of 8-hydroxyquinoline:

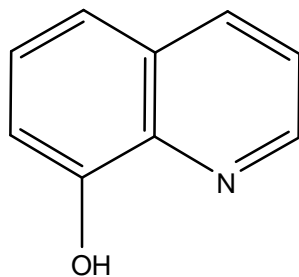


Figure 1. Structure of 8-hydroxyquinoline

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

Derivatives of 8-hydroxyquinoline have been used as tropical antiseptic and internal disinfectants that apparently exhibit low toxicity for humans (Underwood, 1998).

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

Synthesis of 8-hydroxyquinoline copper (II) in this research has been done with a mixing/reaction of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ with 8-hydroxyquinoline (in methanol as solvent) in different ammonia concentrations. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate grey-yellow was obtained, this is 8-hydroxyquinoline copper (II) complex.

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

Methodology

Materials

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ p.a (Merck), 8-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

absorption spectrophotometer (AAS), Whatmann paper.

Procedure

8-hydroxyquinoline Copper (II) synthesis

Becker glass I: An aqueous solution contains of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (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 hours, and precipitate was obtained. Precipitate was filtered and washed by methanol and dried at desiccator until 3 days. Precipitate has grey-yellow colour, this is 8-hydroxyquinoline copper (II) complex. Base data, if concentration of ammonia increases, Cu (II)-8-hydroxyquinoline complex was decreasing obtained, indicated with the colour grey-yellow for the complex is less. Analysis of the complex was done with UV-Vis spectrophotometry, AAS, and FTIR.

Result and Discussion

Synthesis of 8-hydroxyquinoline Copper (II) has been done with mixing/reaction of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ with 8-hydroxyquinoline (in methanol as solvent) in different ammonia concentrations. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate grey-yellow was obtained, this is 8-hydroxyquinoline copper (II) complex.

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Ammonia Variation

An aqueous CuSO_4 solution with ammonia concentration differently has a different result. 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 increases, the result of 8-hydroxyquinoline copper (II) decreases. This is indicated by 8-hydroxyquinoline copper (II) grey-yellow is less. If ammonia concentration increases, the system would be a reaction of Cu^{2+} with 8-hydroxyquinoline to form Tetramine Copper(II). So the 8-hydroxyquinoline copper (II) complex decreases.

Ammonia Effect for 8-hydroxyquinoline copper (II) synthesis

From AAS data, if ammonia concentration increases, the result of 8-hydroxyquinoline copper (II) is

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-hydroxyquinoline.

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. Analysis spectrophotometry UV-Vis has used for establish λ_{max} (nm) sample. This wavelength used to trace λ_{max} (nm) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 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 ($d \rightarrow d$) 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 ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) to 394 nm (8-hydroxyquinoline copper (II) complex) is indicated coordination bonding would be obtained. (Fessenden, 1986).

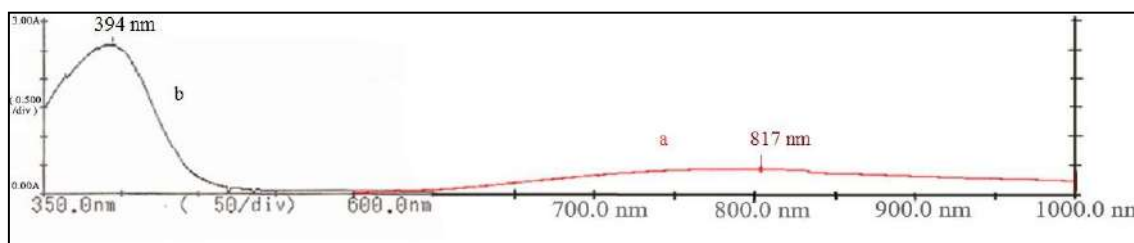


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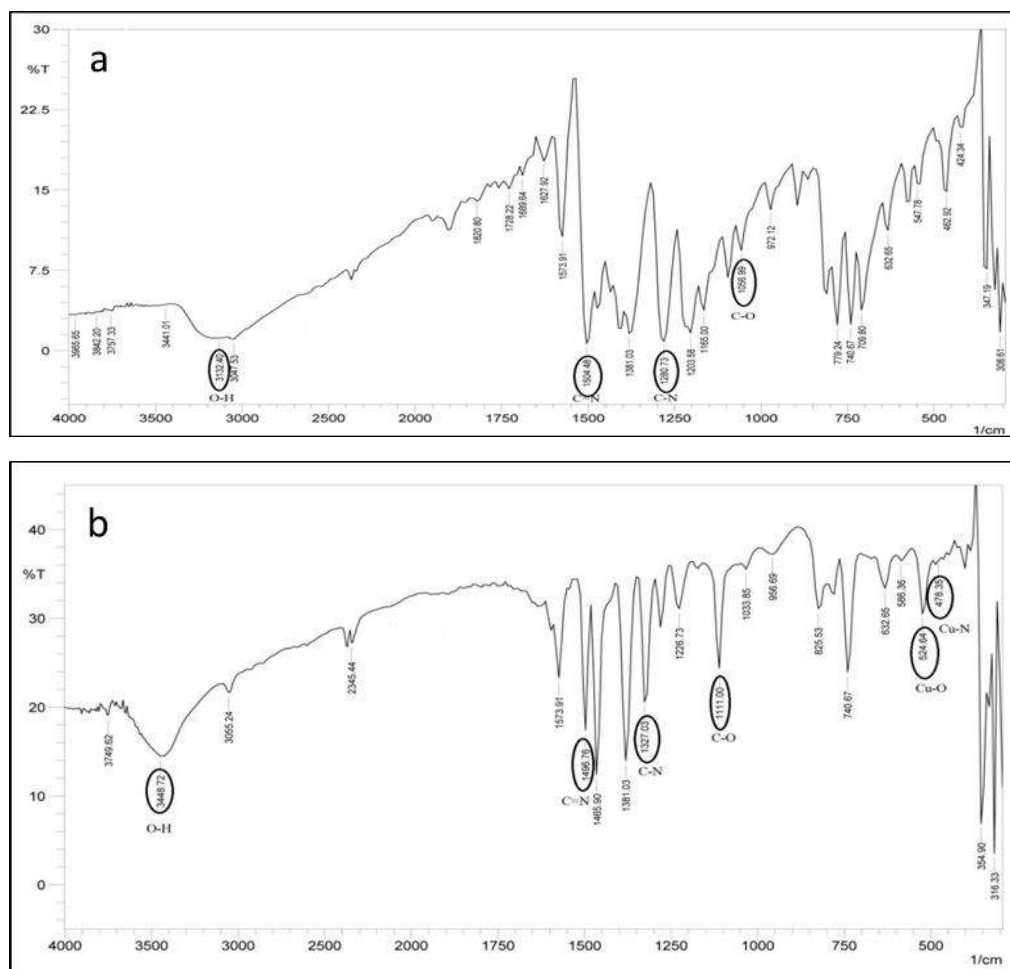


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Conclusion

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

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

by Sriatun Sriatun

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Suhartana, Laelatri Agustina, Sriatun

Abstract

Synthesis and characterization of complex compounds of 8-hydroxyquinoline copper (II) has been done. Synthesis of 8-hydroxyquinoline copper (II) is done by mixing $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 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 obtained decreasing.

Keywords: Synthesis, complex compound Cu- 8-hydroxyquinoline

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Introduction

Oxine or 8-hydroxyquinoline ($\text{C}_9\text{H}_7\text{ON}$) forms sparingly soluble derivatives with metallic ions, which have the composition $\text{M}(\text{C}_9\text{H}_7\text{ON})_2$ if co- coordination number of the metal is four (e.g., magnesium, zinc, copper, cadmium, lead and indium), $\text{M}(\text{C}_9\text{H}_7\text{ON})_3$ if the co- coordination number is six (e.g., aluminium, iron, bismuth, and gallium), and $\text{M}(\text{C}_9\text{H}_7\text{ON})_4$ if the co- coordination number is eight (e.g., thorium and zirconium). There are, however, some exceptions, for examples, $\text{TiO}(\text{C}_9\text{H}_7\text{ON})_2$, $\text{MnO}_2(\text{C}_9\text{H}_7\text{ON})_2$, $\text{WO}_2(\text{C}_9\text{H}_7\text{ON})_2$, and $\text{UO}_2(\text{C}_9\text{H}_7\text{ON})_2$ (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 ($59.6 \times 10^6 \text{ s/m}$) 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 , ^{64}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 al, 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:

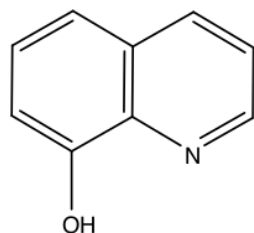


Figure 1. Structure of 8-hydroxyquinoline

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

Derivatives of 8-hydroxyquinoline have been used as tropical antiseptic and internal disinfectants that apparently exhibit low toxicity for humans (Underwood, 1998).

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

Synthesis of 8-hydroxyquinoline copper (II) in this research has been done with a mixing/reaction of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ with 8-hydroxyquinoline (in methanol as solvent) in different ammonia concentrations. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate grey-yellow was obtained, this is 8-hydroxyquinoline copper (II) complex.

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

Methodology

Materials

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 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

absorption spectrophotometer (AAS), Whatmann paper.

Procedure

8-hydroxyquinoline Copper (II) synthesis

Becker glass I An aqueous solution contents of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (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 hours, and precipitate was obtained. Precipitate was filtered and washed by methanol and dried at desiccator until 3 days. Precipitate has grey-yellow colour, this is 8-hydroxyquinoline copper (II) complex. Base data, if concentration of ammonia increases, Cu (II)-8-hydroxyquinoline complex was decreasing obtained, indicated with the colour grey-yellow for the complex is less. Analysis of the complex was done with UV-Vis spectrophotometry, AAS, and FTIR.

Result and Discussion

Synthesis of 8-hydroxyquinoline Copper (II) has been done with mixing/reaction of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ with 8-hydroxyquinoline (in methanol as solvent) in different ammonia concentrations. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate grey-yellow was obtained, this is 8-hydroxyquinoline copper (II) complex.

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Ammonia Variation

An aqueous CuSO_4 solution with ammonia concentration differently has a different result. 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 increases, the result of 8-hydroxyquinoline copper (II) decreases. This indicates that 8-hydroxyquinoline copper (II) grey-yellow is less. If ammonia concentration increases, the system would be a reaction of Cu^{2+} with 8-hydroxyquinoline to form Tetramine Copper (II). So the 8-hydroxyquinoline copper (II) complex decreases.

Ammonia Effect for 8-hydroxyquinoline copper (II) synthesis

From AAS data, if ammonia concentration increases, the result of 8-hydroxyquinoline copper (II) is

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-hydroxyquinoline.

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

$[\text{Cu}^{2+}]$ total	[Ammonia]	$[\text{Cu}^{2+}]$ 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. Analysis spectrophotometry UV-Vis has used for establish λ_{max} (nm) sample. This wavelength used to trace λ_{max} (nm) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and λ_{max} (nm) 8-hydroxyquinoline copper (II) complex, after reaction was occurred.

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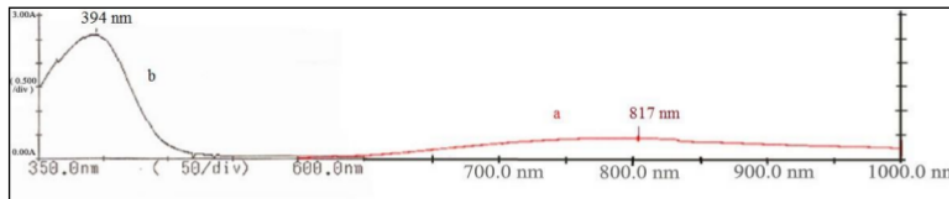


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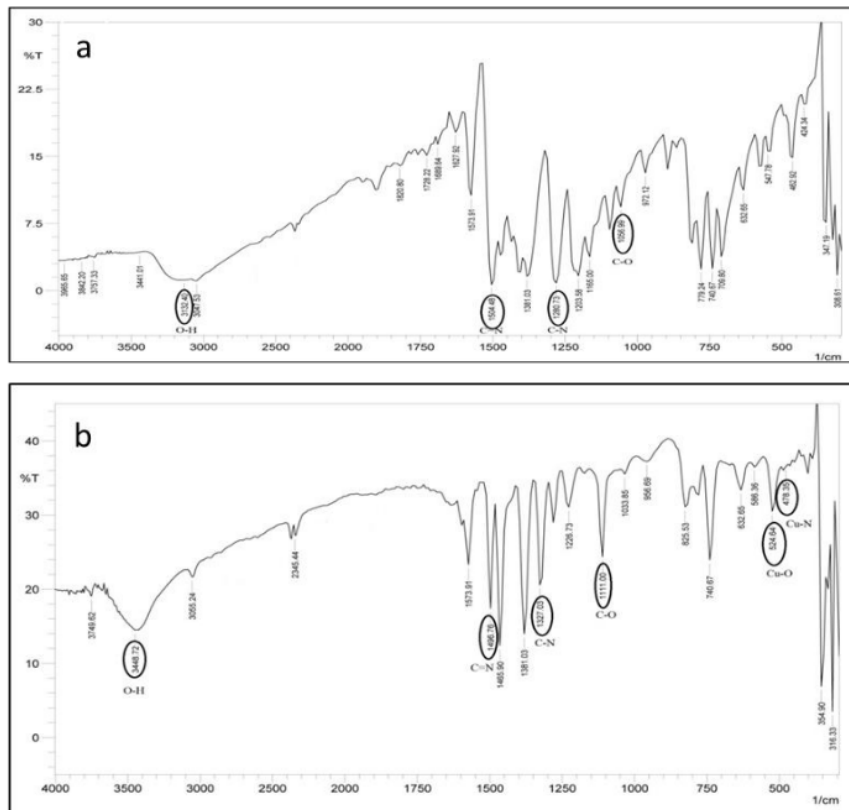


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