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

Judul Karya Ilmiah (Prosiding) Nama/ Jumlah Penulis Status Pengusul	: : :	Influ Suha Penu	ences of Ammonia for Synthe artana, Laelatri Agustina, Sria ilis pendamping	esis tun (	of 8-Hydroxiquinoline Copper(II) (3)
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
			Alamat Artikel	:	https://jcc.undip.ac.id/assets/attachments/JCC9%2
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					Url Turnitin: (10%)
					https://doc-
					pak.undip.ac.id/3033/27/Turnitin29.pdf
		f.	Terindeks di (jika ada)	:	-

Kategori Publikasi Makalah (beri √pada kategori yang tepat) Prosiding Forum Ilmiah Internasional
 Prosiding Forum Ilmiah Nasional

Hasil Penilaian Peer Review :

	Nilai Mak	simal Prosiding	Nilai Akhir		
Komponen Yang Dinilai	Internasional	Nasional	Yang Diperoleh		
a. Kelengkapan unsur isi prosiding (10%)	1,5		1,5		
b. Ruang lingkup dan kedalaman pembahasan (30%)	4,5		4,3		
<ul> <li>Kecukupan dan kemutahiran data/informasi dan metodologi (30%)</li> </ul>	4,5		4,3		
d. Kelengkapan unsur dan kualitas terbitan /prosiding (30%)	4,5		4,4		
Total = (100%)	15,00		14,5		
<b>Penulis Pendamping:</b> (0,4x14,5)/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 dgunakan 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

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

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		e.	Alamat Repository/Web	:	https://jcc.undip.ac.id/24/the-proceeding-of-9th-
					jcc-semarang-2014.conf
			Alamat Artikel	:	https://jcc.undip.ac.id/assets/attachments/JCC9%2
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					https://doc-
					pak.undip.ac.id/3033/27/Turnitin29.pdf
		f.	Terindeks di (jika ada)	:	-

Kategori Publikasi Makalah (beri √pada kategori yang tepat) Prosiding Forum Ilmiah Internasional
 Prosiding Forum Ilmiah Nasional

Hasil Penilaian Peer Review :

	Nilai Mak	Nilai Akhir	
Komponen Yang Dinilai	Internasional	Nasional	Yang Diperoleh
e. Kelengkapan unsur isi prosiding (10%)	1,5		1,2
f. Ruang lingkup dan kedalaman pembahasan (30%)	4,5		4,0
g. Kecukupan dan kemutahiran 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 Pendam	ping: $(0, 4x14)/2 =$	2,8	

### Catatan Penilaian Paper oleh Reviewer :

1. Kesesuaian dan kelengkapan unsur isi prosiding: Unsur isi prosiding lengkap dan sesuai dengan turnitin similarity 18%. Nilai 1,2

### 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

### 3. 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

**4. 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* KARYA ILMIAH : PROSIDING

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			· ·		icc-semarang-2014.conf
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Katagori Dublikagi Makalah			Duosiding Forum Ilm	aiah	Internacional

Kategori Publikasi Makalah (beri ✓ pada kategori yang tepat) *Prosiding* Forum Ilmiah Internasional
 *Prosiding* Forum Ilmiah Nasional

Hasil Penilaian Peer Review :

	Nilai I	Reviewer	
Komponen Yang Dinilai	Reviewer I	Reviewer II	Nilai Rata- rata
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: (0,4x1	(4,25)/2 = 2,85		

Semarang,

Reviewer 1

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

Reviewer 2

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

# CERTIFICATE OF PRESENTATION

Certificate no.: 240/UN7.3.8/2014

Semarang Indonesia

The conference on Green Chemistry

(9)

This is to certify that

# Suhartana

has presented a paper entitled

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

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

Semarang, 13 November 2014

9<sup>th</sup> Joint Conference on Chemistry Chair

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



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Modification of Synthetic Zeolite from Bagasse Ash and Their Characterization (https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev\_Part21.pdf) *Sriatun, Taslimah, Linda Suyati* 

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Calcium Phosphate-Chitosan Composite as a Bone Cement Candidate (https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev\_Part24.pdf) Artike *Tri Windarti and Benjamin Horrocks* 

Artikel 4 negara abstrak terlampir

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The Effect of Coconut Oil Concentration on Physical and Chemical Properties of Cosmetic Emulsions (https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev\_Part34.pdf) *Eni Widiyati, AH. Bambang Setiaji, Totok Eka Suharto, Triyono*  Adsorption of Pb(II) and Co(II) on Adsorbent Clay Immobilized *Saccharomyces cerevisiae* Biomass (https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev\_Part35.pdf) *Fahmiati, Mashuni, L.D. Syahdam Hamidi, Nasra* 

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Zakarias Seba Ngara, I Gusti M. Budiana, Aliwarsito

## Section 3: Analytical Chemistry

Effect of pH on Cu–S TiO<sub>2</sub> Photocatalytic Performance toward Phenol Photodegradation and Cr(VI) Photoreduction by Visible Light Irradiation (https://jcc.undip.ac.id/assets/attachments/JCC9%20– %20content/Proceeding%20content%20rev\_Part47.pdf) *Abdul Haris, Didik Setiyo Widodo, Rahmad Nuryanto* 

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Samuel Budi Wardhana Kusuma, Ibrahim Al Tarawneh, Robert Kreuzig

The Proceeding Of 9th Jcc Semarang 2014 The 13rd Joint Conference on Chemistry JCC-13 2018

Analysis of Nitrosodiethylamine (NDEA) in Salted Fish with Hollow Fibre-Liquid Phase Microextraction Gas Chromatography Flame Ionization Detector(HF-LPME-GC-FID) Method (https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev\_Part57.pdf) *Usreg Sri Handajani, Ganden Supriyanto, Yanuardi Raharjo, Gunawan Dwi Saputra* 

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The Effect of Oil Types on The Characteristics of Solid Soap (https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev\_Part63.pdf) *Mardiyah Kurniasih, Purwati, Anung Riapanitra, Zusfahair, Tri Wahyuni* 

Proceedings of The 9<sup>th</sup> Joint Conference on Chemistry

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

Atyaf Khalid Hammed<sup>a</sup>, Nugroho Dewayanto<sup>a</sup>, D. Dongyun<sup>b</sup>, Mohd Ridzuan Nordin<sup>a</sup>

### Abstract

Nano scale zero-valent iron (NZVI) supported on mesoporous silica (MSN) was synthesized through liquid phase reduction route. in this method, FeCl<sub>3</sub>.6H<sub>2</sub>O solution was titrated with NaBH₄ 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<sup>2</sup>/g to 125.8745 m<sup>2</sup>/g. Preparation of NVZI supported by MSNs was conducted in alkaline solution. Amount of FeCl<sub>3</sub>.6H<sub>2</sub>O in aqueous solution and activated silica in certain ratio was mixed at room temperature. NaBH<sub>4</sub> solutionwas added to the mixture in drop wise manner (3 ml/min) with vigorous stirring at room temperature. NVZI/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 FeCl<sub>3</sub>.6H<sub>2</sub>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, hetero generous catalysis, and separation. Furthermore, scientists are still targeting new adsorbent with good property. in recent years, with the development of nanotechnology, various

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Proceedings of The 9<sup>th</sup> Joint Conference on Chemistry

### **Calcium Phosphate-Chitosan Compositeas a Bone Cement Candidate**

### Tri Windarti<sup>a</sup> and Benjamin Horrocks<sup>b</sup>

### 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 ofNa<sub>2</sub>HPO<sub>4</sub> 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 it 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 applicated 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- $\beta$ -D-Glucose) and has similarity with collagen structure. Chitosan has been widely used in medical field because of it 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.

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# Catalytic properties of bimetallic NiNP-M/AIOH (M = Sn, In, Ga, Ag, Nb, andZr) on selective hydrogenation of furfural

Rodiansono<sup>a</sup>\*, M. D. Astuti<sup>a</sup>A. Ghofur<sup>b</sup>, Shogo Shimazu<sup>c</sup>

### Abstract

A series of bimetallic NiNP-M/AIOH (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/AIOH) 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, N<sub>2</sub>adsorption, and H<sub>2</sub>-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 <sup>1</sup>H 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/AIOH.  $H_2$ -uptake of Ni-M/AIOH also was lower than that of NiNP/AIOH. NiNP-Sn/AIOH and NiNP-In/AIOH 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/AIOH and Ni-In/AIOH catalysts due to the formation of Ni-Sn or Ni-In alloys. On the other hand, NiNP-M/AIOH (M= Ga, Nb, Ag, Zr, and Ga) catalysts showed lower the catalytic activity than that of NiNP/AIOH catalyst. Therefore, further investigation of role of the second metal on the catalytic properties of NiNP-M/AIOH (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  $H_2$ 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  $Cr_2O_3$  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-promotors were applied such as Cu [4, 8], Fe, Ce [5-7],

and Sn [10] in order to improve its chemoselectivity towards C=Orather 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/SiO<sub>2</sub> 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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# Synthesis and Characterization of Nano Scale Zero-Valent Iron Supported on Mesoporous Silica

Atyaf Khalid Hammed<sup>a</sup>, Nugroho Dewayanto<sup>a</sup>, D. Dongyun<sup>b</sup>, Mohd Ridzuan Nordin<sup>a</sup>

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Nano scale zero-valent iron (NZVI) supported on mesoporous silica (MSN) was synthesized through liquid phase reduction route. in this method, FeCl<sub>3</sub>.6H<sub>2</sub>O solution was titrated with NaBH₄ 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<sup>2</sup>/g to 125.8745 m<sup>2</sup>/g. Preparation of NVZI supported by MSNs was conducted in alkaline solution. Amount of FeCl<sub>3</sub>.6H<sub>2</sub>O in aqueous solution and activated silica in certain ratio was mixed at room temperature. NaBH<sub>4</sub> solutionwas added to the mixture in drop wise manner (3 ml/min) with vigorous stirring at room temperature. NVZI/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 FeCl<sub>3</sub>.6H<sub>2</sub>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

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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).

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

### Suhartana, Laelatri Agustina, Sriatun

### 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 8hydroxyquinoline 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 cocoordination 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 dshells, 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. <sup>63</sup>Cu and <sup>65</sup>Cu are stable, with <sup>63</sup>Cu comprising approximately 69 % of naturally occurring copper. The other isotopes (<sup>62</sup>Cu, <sup>63</sup>Cu and <sup>67</sup>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 antiamoebic agents, 8- hydroxyquinoline behaves as bidentate (N & O<sup>-</sup>) 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<sup>-</sup>) 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 8hydroxiquinoline 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 8hydroxyquinoline used as ligand. 8- hydroxyquinoline ligand has O at CO bonding and N as electron donor [Huheey, 1981]. So bonding Cu metals and 8hydroxyquinoline ligand has been obtained. "Continue Variation" method has done to form 8hydroxyquinoline copper (II) (Sugiarto, 2009).

Synthesis 8- hydroxyquinoline copper (II) this research has done with mixing/ reaction CuSO<sub>4</sub>.5H<sub>2</sub>O with 8hydroxyquinoline (in methanol as solvent) in different ammonia concentration. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate greyyellow 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<sub>4</sub>.5H<sub>2</sub>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 absorptions spectrophotometer (AAS), Whatmann paper.

### Procedure

### 8-hydroxyquinoline Cuprum (II) synthesis

Becker glass I An aqueous solution contents of CuSO<sub>4</sub>.5H<sub>2</sub>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 aguadest, Becker glass II Have contents 8- hydroxyguinoline 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 8hdroxyquinoline 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<sub>4</sub>.5H<sub>2</sub>O 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.

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### 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) greyyellow is less. If ammonia concentration is increase, the system would be reaction  $Cu^{2+}$  with 8hydroxiquinoline 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

Green Chemistry Section 1: Material Chemistry, Suhartana, et al. This Proceedings©Chemistry Department, FSM, Diponegoro University 2015 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<sup>2+</sup> at Variation Ammonia concentration

[Cu <sup>2+</sup> ] total	[Ammonia]	[Cu <sup>2+</sup> ] remainder
100 ppm	0.01 M	3.4 ppm
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### UV-Vis characterization

The adsorption and assignments related the ligand and the complex showed in Figure 2. Analysisspectrophotometry UV-Vis has used for establish  $\lambda_{max}$  (nm) sample. This wavelength used to trace  $\lambda_{max}$  (nm) CuSO<sub>4</sub>.5H<sub>2</sub>O and  $\lambda_{max}$  (nm) 8-hydroxyquinoline copper (II) complex, after reaction was occurred.

The spectra where the electronic configuration of the metal d<sup>10</sup>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<sub>4</sub>.5H<sub>2</sub>O) to 394 nm (8-hydroxyquinoline copper (II) complex) is indicated coordination bonding would be obtained. (Fessenden, 1986).



Figure 2Spectra UV-VIS (a). CuSO<sub>4</sub>.5H<sub>2</sub>O, (b). 8-hydroksikuinolin copper (II) at methanol solvent

Transition electronic Energy 10Dq from CuSO<sub>4</sub>.5H<sub>2</sub>O 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  $\lambda_{max}$  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<sub>4</sub>.5H<sub>2</sub>O

 and 8-hydroxyquinoline cuprum (II) complex

No.	Compound	λ <sub>max</sub> (nm)	10Dq (KJ/mol)
1.	CuSO <sub>4</sub> .5H <sub>2</sub> O	817	146,288
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### **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 <sup>-1</sup> )	8 -hydroxyquinoline copper (II). (cm <sup>-1</sup> )
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The spectrum of phenol exhibited the strong band of 1056,9 cm<sup>-1</sup> for 8-hydroxy-quinoline and 1111 cm<sup>-1</sup> for 8 -hydroxyquinoline copper (II). While another strong adsorption bands at 1280 cm<sup>-1</sup> for 8-hydroxyquinoline and 1327 cm<sup>-1</sup> for 8 -hydroxyquinoline copper (II).

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stretching vibration of V (C-O) of the carbonyl group have been found in the range between (1100 cm<sup>-1</sup>-1200 cm<sup>-1</sup>). 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  $\rm cm^{-1}\text{-}1350~\rm cm^{-1}$  shifted to the higher frequencies. Adsorption assigned for V (Cu-N) was

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### Conclusion

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

- 1. CuSO<sub>4</sub> 5 H<sub>2</sub>O has UV- Vis characteristic adsorption at  $\lambda_{max}$  817 nm, and 8- hydroxyquinoline (8HQ) copper (II) has UV-Vis characteristic adsorbtion at  $\lambda_{max}$  394 nm.
- 8-hydroxyquinoline would coordinate bond with Cu<sup>2+</sup> ion, by N atomic and O atomic.

### Acknowledgments

I would like to thank my student laelatri Agustina, also my friend Sri Atun for her support during my experiment. You have helped so much, there is no way I could possibly ever repay you.

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

by Sriatun Sriatun

Submission date: 25-Apr-2020 11:06PM (UTC+0700) Submission ID: 1307518353 File name: es\_of\_Ammonia\_for\_Synthesis\_of\_8-Hydroxiquinoline\_Copper\_II.pdf (663.85K) Word count: 2164 Character count: 12264

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

Suhartana, Laelatri Agustina, Sriatun

### 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 8hydroxyquinoline 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 (C<sub>9</sub>H<sub>7</sub>ON) 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<sub>9</sub>H<sub>7</sub>ON)<sub>4</sub> if the co-coordination number is eight (e.g., thorium and zirconium). There are, however, some exceptions, for examples, TiO (C<sub>9</sub>H<sub>7</sub>ON)<sub>2</sub>, MnO<sub>2</sub> (C<sub>9</sub>H<sub>7</sub>ON)<sub>2</sub>, wO<sub>2</sub> (C<sub>9</sub>H<sub>7</sub>ON)<sub>2</sub>, and UO<sub>2</sub>(C<sub>9</sub>H<sub>7</sub>ON)<sub>2</sub> (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 dshells, 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. <sup>63</sup>Cu and <sup>65</sup>Cu are stable, with <sup>63</sup>Cu comprising approximately 69 % of naturally occurring copper. The other is copes (<sup>62</sup>Cu, <sup>63</sup>Cu and <sup>67</sup>Cu) are radioactive. Copper is present in the earth crust at a conc 6 tration 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 (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 antiamoebic 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:

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

Synthesis 8- hydroxyquinoline copper (II) this research has done with mixing/ reaction CuSO<sub>4</sub>.5H<sub>2</sub>O with 8hydroxyquinoline (in methanol as solvent) in different ammonia concentration. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate greyyellow 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 0 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

**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 Proceedings of The 9<sup>th</sup> Joint Conference on Chemistry

absorptions spectrophotometer (AAS), Whatmann paper.

### Procedure

### 8-hydroxyquinoline Cuprum (II) synthesis

Becker glass I An aqueous solution contents of CuSO<sub>4</sub>.5H<sub>2</sub>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 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 8hdroxyguinoline 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<sub>4</sub>.5H<sub>2</sub>O with 8hydroxyquinoline (in methanol as solvent) in different ammonia concentration. Mixing by magnetic stirrer. Result obtained dried by desiccator, precipitate greyyellow 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 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) greyyellow is less. If ammonia concentration is increase, the system would be reaction  $Cu^{2+}$  with 8hydroxiquinoline to form Tetramine Copper(II). So the 8-hydroxiquinoline copper (II) complex is decrease.

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