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

Judul Karya Ilmiah (Prosiding)	:	Influences of Ammonia for Synthesis of 8-Hydroxiquinoline Copper(II)						
Nama/Jumian Penulis	•	Suna	Sunanana, Laciau Agustina, Shatun (5)					
Status Pengusul	:	Penulis pendamping						
Identitas Prosiding :		a.	JudulProsiding	:	Green Chemistry: Proceeding of 9 th Joint Conference on Chemistry, 12-13 November 2014			
		b.	ISBN/ISSN	:	978-602-285-049-6			
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		d.	Penerbit/Organiser		UNNES Press			
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		f.	Terindeks di (jika ada)	:	-			
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Hasil Penilaian Peer Review:

	Nilai Mak	Nilai Akhir	
Komponen Yang Dinilai	Internasional	Nasional	Yang Diperoleh
a. Kelengka pan unsur isi prosiding (10%)	1,5		1,5
b. Ruang lingkup dan kedalaman pembahasan (30%)	4,5		4,3
c. Kecukupan dan kemutahiran 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 Pendam	ping: (0,4x14,5)/2 =	=2,9	

Catatan Penilaian Paper oleh Reviewer :

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- 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 metodo lgin ya cukup memadai. Nila i 4,3
- 4. Kelengkapan unsur dan kualitas terbitan/prosiding: Unsur prosiding lengkap, kualitas prosiding baik. Nilai 4,4

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

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

Judul Karya Ilmiah (Prosiding)	:	Influences of Ammonia for Synthesis of 8-Hydroxiquinoline Copper(II)					
Nama/Jumlah Penulis	:	Suhartana, Laelatri Agustina, Sriatun					
Status Pengusul	:	Penulis pendamping					
Identitas Prosiding :		a. Judul Prosiding	:	Green Chemistry: Proceeding of 9 th Joint Conference on Chemistry, 12-13 November 2014			
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		Alamat Artikel	:	https://jcc.undip.ac.id/assets/attachments/JCC9%2			
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				s of Ammonia for Synthesis of 8-			
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		f. Terindeks di (jika ada)	:	-			
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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 Pendan	nping: $(0, 4x14)/2 =$	2,8	

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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 a dalah sintesis tembaga 8-hidroksi kuinolin dan pengaruh ammonia pada riset tersebut. Keda laman pembahasan kurang didukung oleh referensi terkait. Nila i 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

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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

Judul Karya Ilmiah Nama/ Jumlah Penulis Status Pengusul	: Inf : Sub : Pen	luences of Ammonia for Syntl nartana, Laelatri Agustina, Sria nulis pendamping	hesis atun	s of 8-Hydroxiquinoline Copper(II)
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	f.	Terindeks di (jika ada)	:	-

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

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Hasil Penilaian *Peer Review* :

	Nilai I		
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
 Kecukupan dan kemutahiran data/informasi dan metodologi (30%) 	4,3	4,3	4,3
 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		

Reviewer 2

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

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12-13 November 2014 Semarang, Indonesia

Proceedings of The 9th Joint Conference on Chemistry

Diponegoro University (UNDIP), Semarang State University (UNNES), Sebelas Maret University (UNS) and Jenderal Soedirman University (UNSOED)

Grand Candi Hotel, Semarang, 12-13 November, 2014

Green Chemistry

Editors

Dwi Hudiyanti Agustina L.N. Aminin Adi Darmawan Yayuk Astuti

> UNNES Press 2015

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Preface to The Conference Proceedings

We are very pleased to introduce The 9th Joint Conference on Chemistry (9th JCC) held by Diponegoro University (UNDIP) On behalf of the Chemistry Consortium in Central Java, Indonesia. The JCC is an annual conference organized by the consortium of Chemistry Department of four universities in Central Java: Diponegoro University (UNDIP), Semarang State University (UNNES), Sebelas Maret University (UNS) and Jenderal Soedirman University (UNSOED); since 2006. The growing of environmental problems that persist to escalate worldwide has compelled us to select "Green Chemistry" as the leading theme of the 9th JCC.

We had 10 plenary speakers, 10 invited speakers and over 120 suitable papers from 11 countries were submitted for presentation at the conference. This required the program to be organized in five parallel sessions, each on a specific theme, to provide each paper with sufficient time for presentation and to accommodate all of them within the overall time allocated. One of the five sessions contained analytical chemistry. A second session was devoted to the theme of biochemistry. The third and fourth session were dedicated to physical and material chemistry. The fifth session was concerned with chemical education. These were well represented in the program of the conference and were clearly topics which continue to stimulate a global interest. The programs were chaired in a professional and efficient way by the session chairmen who were selected for their international standing in the subject.

All the papers went through a peer-review procedure prior to being accepted for publication in this book. These Proceedings present the permanent documentation of what was presented. They indicated the state of advancement at the time of writing of all aspects of this theme and will be very useful to all people in the field.

As a final point, it is appropriate that we record our thanks to our fellow members of the steering committee, organizing committee, and scientific committee. We are also indebted to those who served as chairmen. Without their support, the conference could not have been the success that it was. We also would like to express our sincere gratitude to all authors for their valuable contributions. We are thankful to the students of Chemistry Department Faculty of Science and Mathematics Diponegoro University especially to Maya and Fuad for their support during preparation of the manuscript.

Dwi Hudiyanti Agustina L.N. Aminin Adi Darmawan Yayuk Astuti

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Prof. Keiji Tanaka, Department of Applied Chemistry, Kyushu Univers<mark>ity, Japan</mark> Topic of the talk: "Physical Properties of Polymer Solids: from bulk to surface'



Prof. Hiroshi Kitagawa, Graduate School of Science, Kyoto University, Japan Topic of the talk: "Functional Materials on the Basis of Elemental Strategy"

Dr. David G. Churchill, Department of Chemistry, Korea Advanced Institute of Science and Technology (KAIST), South Korea Topic of the talk: Heavier Group 16-based Organic Molecular Probes for Neurodegenerative Disease Research



Assoc. Prof. Xu Qing-Hua, Department of Chemistry, National University of **Singapore** Topic of the talk: "Plasmon coupling enhanced two-photon photoluminescence and

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their applications"



Prof. Hitoshi Miyasaka, Institute of Material Research

(IMR), Tohuku University, Japan



Topic of the talk: "The design of Functional Metal-Organic Frameworks toward the Synergistic Control of Electron, Spins, Structure and Chemical Interactions"



Prof. Nasser Safari, Department of Chemistry, Shahid Beheshti University<mark>, Iran</mark> Topic of the talk: "Porphyrin-based Photosensitizers for Green Oxidation of Substrates with Molecular Oxygen and Sunlight"

Prof. Pimchai Chaiyen, Department of Biochemistry/ Center for Excellence in Protein Structure and Function, Mahidol University<mark>, Thailand</mark>





Dr. M. Abdul Kadir Martoprawiro, Chemistry Department, Bandung Institute of Technology and Chair of Indonesian Chemical Society

Dr. Edy Giri Rachman Putra, Neutron Scattering Laboratory. National Nuclear Energy Agency of Indonesia (BATAN)



Invited Speakers:

Prof. Farook Adam, University of Science Malaysia
Prof. Mohd Marsin Sanagi, Universiti Teknologi Malaysia
Dr. Dwi Hudiyanti, Diponegoro University, Indonesia
Dr. Khairul Anam, Diponegoro University, Indonesia
Dr. Triana Kusumaningsih, Sebelas Maret University, Indonesia
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Prof. Supartono, Semarang State University, Indonesia
Prof. Kasmadi Imam Supardi, Semarang State University, Indonesia

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, FeCl₃.6H₂O 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 m²/g to 125.8745 m²/g. Preparation of NVZI supported by MSNs was conducted in alkaline solution. Amount of FeCl₃.6H₂O in aqueous solution and activated silica in certain ratio was mixed at room temperature. NaBH₄ 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 FeCl₃.6H₂O 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

Green Chemistry Section 1: Material Chemistry, Atyaf Khalid Hammed, et al. This Proceedings©Chemistry Department, FSM, Diponegoro University 2015 nanomaterials have been extensively used in adsorption (Shih & Tai., 2010).

The removal of nanosized adsorbents is an important issue and requires high cost. The application of magnetic particles have been proposed as an approach to imparting magnetic properties onto adsorbents (Btatkan et al., 2013), which can be separated from treated water by a simple magnetic process. Ferriferous salts particles have become the most popular magnetic material due to its low cost, low toxicity and eco-friendliness (Cho et al., 2013). The correlation of equilibrium data using either a theoretical or empirical equation is essential for adsorption data interpretation and prediction. Several mathematical models can be used to describe experimental data of adsorption isotherms (Sun et al., 2014). in this work, the fume silica was activated for the preparation of MSN, followed by impregnation of the ferric chloride hexahydrate (FeCl₃.6H₂O) onMSN. This NVZI/MSN nano composite then used as highly efficient adsorbent with excellent separation properties. Methylene blue (MB) was selected as model pollutant to evaluate the optimum adsorption condition included contact time, initial pH solution, temperature and adsorbent dose under laboratory conditions.

Methodology

Sodium borohydride (NaBH₄) 98.5%, iron (III) chloride hexahydrate (FeCl₃·6H₂O) 99% and methylene blue (MB) were obtained from Merck. Fumed micro porous silica (purity 99%, Dongyang Chem. Co) with a median particle size of 2.5 μ m were used as the silicate source for the synthesis of mesoporous silica. Sodium hydroxide (NaOH) and hydrochloric acid (HCl) were obtained from Sigma-Aldrich, while analytical grade absolute ethanol was obtained from Merck and used without purification.

Production of NZVI involved the reduction of FeCl₃.6H₂O in aqueous solution by NaBH₄ (Idris et al., 2007). The method comprised of four stages which were mixing, separating, washing and drying. These two chemicals were mixed by dripping 1 M of NaBH₄ solution into 50 ml of 2 M FeCl₃.6H₂O continuously while stirring the reaction mixture well. Excess NaBH₄ was typically applied in order to accelerate the reaction and ensured uniform growth of iron particles. Immediately after the addition of the first drop of reducing agent into iron solution, black particles appeared. The maximum yield of black iron particles was obtained by further mixing for 15 to 20 min. Black iron particles were then separated from the solution by vacuum filtration using Whatmann cellulose nitrate membrane filter (1 µm).

Three grams of fumed silica was dissolved in 50 ml concentrated hydrochloric acid and refluxed for 3 hours. A white gel was then formed. The reaction

mixture was left to cool naturally to room temperature, and then filtered with the filtrate washed with deionized water several times until the pH 7 was achieved. The sample was dried at 150 °C for 4 h and kept in desiccators for use.

One gram of dry MSN was equilibrated with 30 ml 1.0 M ferric chloride solution for 3 h. The slurry was diluted using a mixture of ethanol and deionized water at ratio 3:1(v/v), then 50 mL of 0.2 M NaBH₄ was added in a drop wise manner (3.0 ml min⁻¹) into the slurry at 25 °C with magnetic stirring. After 30 min of agitation, the (NZVI/MSNs), nanocomposite were separated from the mixture and washed with acetone for three times and then vacuum dried at 60 °C and stored in a desiccator for further use.

The diffractogram of adsorbents were obtained by a Rigaku miniFlex II desktop X-ray Diffractometer with Cu K α as a source at a tube voltage of 30 kV and a current of 15 mA. The diffractogram patterns were collected in 20 range from 0° to 80° with step sizes of 0.02° and at a scanning speed of 1°/min. The surface area of adsorbent was determined from the adsorption isotherms of nitrogen at -196 °C onto the catalyst using Micromeritics ASAP 2000. All the samples were degassed at 105 °C prior to the analysis and the adsorption of N_2 was measured at -196 °C. Brunauer-Emmett-Teller (BET) equation was employed to calculate the specific surface area. The surface structure of adsorbent was observed by using FESEM (JEOL). FTIR spectra were collected by using PerkinElmer Spectrometry 100.

Batch adsorption experiments were conducted by using aqueous solution of methylene blue. The dve stock solution was prepared by dissolving accurately weighted methylene blue in distilled water to the concentration of 500 mg L⁻¹. The experimental solutions were obtained by diluting the dye stock solution in accurate proportions to required initial concentrations. А Genesys 105 UV-VIS spectrophotometer was used for the measurement of absorbance at the predetermined maximum absorbance wavelength (λ_{max} = 663 nm) of the MB dye to determine the concentrations of MB in the solution.

Batch adsorption experiments were carried out to study the different parameters included contact time, pH of solution, temperature, and initial concentration of methylene blue. Amount of NZVI/MSN was loaded into MB solution in 250 ml flask sealed with rubber stopper, then placed on a rotary shaker at 150 rpm during the entire experiment period. Equilibrium time of adsorption was determined by using 0.1 g of NZVI/MSN in 100 ml of 15 mg/L MB solutions. The mixture was equilibrated by shaking thoroughly for different time intervals viz. 2.5, 5, 10, 20, 40, 60 90.120 and 180 min. At the end of the shaking period, the reaction solution was sampled by a syringe. The sample was then filtered immediately through 0.22 µm

Calcium Phosphate-Chitosan Compositeas a Bone Cement Candidate

Tri 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 ofNa₂HPO₄ 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- β -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.

Methodology

Powder Preparation

Dicalcium Hydrogen Phosphate (DHP, CaHPO₄). A slow reaction of 250 mL CaCl₂ 1M with 250 mL Na₂HPO₄ (Merck, Darmstadt, Germany) 1M was conducted for 24 hours with gentle stirring.

Calcium Phosphate (CP). CP was synthesized by solid state reaction. Pre-synthesized DHP and calcium carbonate (CaCO₃) (Merck, Darmstadt, Germany) were used as reactant. Ten grams of CaCO₃ and 27.2 g of CaHPO₄ were dispersed in ethanol for 4 hours by gentle stirring to homogeneous the reactants. Powder was dried by decantation and evaporation of ethanol. After that dry powder was milled with mortar and sieved for 230 mesh. Solid state reaction was conducted at 1200 °C for 15 hours. The product was removed from furnace, quenched in air, grounded in a mortar and passed a 230 mesh sieve.

Production of Calcium Phosphate Cement

Calcium phosphate from previous work was used as powder phase and aqueous solution of 2.5 % $Na_2HPO_4.2H_2O$ (Merck, Darmstadt, Germany) (dissolved in DI water) was used as liquid phase. Powder phase and liquid phase were mixed on a glass plate by gentle mixing using spatula until a viscous paste formed. Paste then dried at room temperature on an open air. The liquid/powder ratio (L/P) was varied at 0.5 – 0.65 (v/m).

Production of Calcium Phosphate-Chitosan Composite

Calcium phosphate-chitosan composite was produced by adding chitosan (medium molecular weigth, Iceland Aldrich) to the liquid phase with L/P ratio =1. The concentration of chitosan was varied at 1-5% (w/w).

Material Characterization

The functional groups were investigated by Fourier Transform Infra Red (FTIR, Shimadzu). X-ray diffraction (XRD, Shimadzu) analyses were performed for phase analysis of powder phase, CPC and CPC-chitosan composite. The scanning range of sample was 10-80° and scanning speed 2°/min. The morphology and chemical composition were examined with Scanning Electron Microscope - Energy Dispersive Spectroscopy (SEM-EDS, JEOL/EO JSM-6510)

Results and Discussion

CP compounds as powder phase was synthesized by solid state reaction method. The expected reaction mechanism can be described as follow: high thermal energy will cause excitation of vibrational motion of covalent bonds in reactants so that simple gases such as CO₂, H₂O and H₂ will be released. The thermal energy then will drive PO_4^{3-} ions to diffuse through reactant interface and fill the hole in CaHPO₄ and CaCO₃ crystal structure and form calcium phosphate compound with Ca/P = 1.5.

From FTIR spectra (Figure 1), specific peaks that indicated calcium phosphate compounds were detected.Peak at 956.69 cm⁻¹ and 472.58 cm⁻¹confirms the present of $v_1 PO_4^{3-}$ and $v_2 PO_4^{3-}$ vibration. for v_3 vibration mode, peaks appear at 1049.28 and 1089.78 cm⁻¹, and v_4 vibration appear at 563.21 and 603.72 cm⁻¹. Adsorption of IR light at 3568.31 and 3641.60 cm⁻¹ indicated OH bond. The presence of CO_3^{2-} was detected at wave number of 1419.61 and 1456.90 cm⁻¹. This result was closed to Vascoccellos and Santos (Vascoccellos and Santos, 2013) research data.



Figure 1. The FTIR spectra of powder phase

XRD diffractogram of powder phase is given in Figure 2. The XRD pattern exhibited characteristic peaks of hydroxyapatite [HA, $Ca_{10}(PO_4)_6(OH)_2$] (JCPDS card no. 09-0432) as primary product and α -Tricalcium phosphate [α -TCP, $Ca_3(PO_4)_2$](JCPDS card no. 06-0200) as secondary product. by Scherer equation confirmed that crystallite size of HA and α -TCP are 60.11 nm and 53.06 nm, respectively.



Figure 2. The XRD diffractogram of powder phase

Powder phase was mixed with liquid phase to produce viscous paste. The presence of Na_2HPO_4 in liquid phase

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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. ⁶³Cu and ⁶⁵Cu are stable, with ⁶³Cu comprising approximately 69 % of naturally occurring copper. The other isotopes (⁶²Cu, ⁶³Cu and ⁶⁷Cu) 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⁻) 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 CuSO₄.5H₂O 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

CuSO₄.5H₂O 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 CuSO₄.5H₂O (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 CuSO₄.5H₂O 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) 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 Cu²⁺ 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) CuSO₄.5H₂O and λ_{max} (nm) 8-hydroxyquinoline copper (II) complex, after reaction was occurred.

The spectra where the electronic configuration of the metal d¹⁰continuous 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 (CuSO₄.5H₂O) to 394 nm (8-hydroxyquinoline copper (II) complex) is indicated coordination bonding would be obtained. (Fessenden, 1986).



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Table 5: IR spectra

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Conclusion

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

- 1. CuSO₄ 5 H₂O 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.
- 8-hydroxyquinoline would coordinate bond with Cu²⁺ 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

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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- hydroxyquioline copper (II) is done by mixing CuSO₄5H₂O 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-(s)low. Characterization of complex compound was done by analysis of UV-Vis, AAS and FTIR. US 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 (C₉H₇ON) forms sparingly soluble derivatives with metallic ions, which have the composition $M(C_9H_7ON)_2$ i 3 p- 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., alun Sjum, 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 (C₉H₇ON)₂, MnO₂ (C₉H₇ON)₂, WO₂ (C₉H₇ON)₂, and UO₂(C₉H₇ON)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. ⁶³Cu and ⁶⁵Cu are stable, with ⁶³Cu comprising approximately 69 % of naturally occurring copper. The other is the coper (⁶²Cu, ⁶³Cu and ⁶⁷Cu) 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 (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 CuSO₄.5H₂O 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 9th Joint Conference on Chemistry

absorptions spectrophotometer (AAS), Whatmann paper.

Procedure

8-hydroxyquinoline Cuprum (II) synthesis

Becker glass I An aqueous solution contents of CuSO₄.5H₂O (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 CuSO₄.5H₂O 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-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 Cu²⁺ 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 Cu²⁺ ions to react with 8-hydroxiquinoline.

Table 1.Concentration Cu²⁺ 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

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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 λ_{max} (nm) sample. This wavelength used to trace λ_{max} (nm) CuSO4.5H₂O and λ_{max} (nm) 8-hydroxyquinoline copper (II) complex, after reaction was occurred.

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