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The INTERNATIONAL CONFERENCE ON CHEMISTRY AND MATERIAL SCIENCE (IC2MS) 2017

"Advancing to the frontier of innovation in chemistry and material science"

4-5 November 2017 IJEN SUITES RESORT & CONVENTION MALANG, INDONESIA



PREFACE

On behalf of the Organizing Committee, I would like to welcome all of you, keynote speakers, invited speakers, distinguished guests, and participants, to the **International Conference on Chemistry and Material Science (IC2MS) 2017**. This conference is organized by Chemistry Department, Brawijaya University, and is held for the first time. The IC2MS runs for 2 days, 4-5 November 2017, and consists of 5 mini symposia, they are:

- Analytical and Inorganic Chemistry
- Biochemistry and Organic Chemistry
- Synthetic and Catalyst Materials, Renewable Energy and Fuel Cells, Molecular Science & Computational Chemistry
- Materials Characterization
- Nanomaterials & Nanodevices and Physical Chemistry

The accepted papers of the conference will be published in IOP Conference Series: Materials Science and Engineering. Around 200 people are attending this conference. They consist of 150 presenting participants, 50 non-presenting participants, 5 keynote speakers, and 6 invited speakers. In terms of country of origin, the participants of the IC2MS are coming from 8 countries, including Indonesia, Brunei Darussalam, Thailand, Taiwan, Japan, Kazakhstan, Australia, and Singapore.

The IC2MS should bring advantages for all participants. The IC2MS attendees can exchange and share their experiences and research results on all aspects of chemistry and material sciences. The IC2MS also provides a premier interdisciplinary platform for researchers, practitioners and educators to present and discuss the most recent innovations, trends, and concerns as well as practical challenges encountered and solutions adopted in the fields of chemistry and material sciences.

The ICM2S could not become a reality without the help and assistance of many parties. Thus, in this occasion I would like to sincerely thank the Rector of Brawijaya University, Dean of Faculty of Mathematics and Natural Sciences Brawijaya University, Head of Chemistry Department, Brawijaya University, all members of the Organizing Committee, Ijen Suites Hotel, and all sponsors, who have provided meaningful help and assistance for the implementation of this conference.

Malang, 4 November 2017 Chairman of the Organizing Committee **Anna Safitri, Ph.D**

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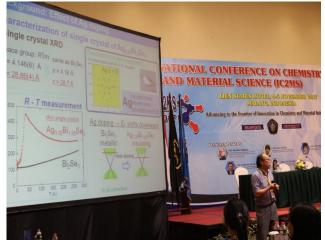
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Table of contents

Volume 299

2018

◆ Previous issue Next issue ▶

International Conference on Chemistry and Material Science (IC2MS) 2017 4–5 November 2017, Malang, East Java, Indonesia

Accepted papers received: 10 January 2018 Published online: 07 February 2018

Open all abstracts

Preface

OPEN ACCESS International Con	nference on Chemis	stry and Material Science (IC2MS) 2017	011001
+ Open abstract	View article	PDF	
OPEN ACCESS			011002
Peer review state			
+ Open abstract	View article	🔁 PDF	
Papers			

ANALYTICAL CHEMISTRY

Determination of glucose in human urine by cyclic voltammetry method using gold electrode

Riyanto and Muh. Supwatul Hakim

+ Open abstract 🔄 View article 🔁 PDF

Peat Water Purification by Hydroxyapatite (HAp) Synthesized from Waste Pensi (<i>Corbicula moltkiana</i>) Shells Matlal Fajri Alif, Wandha Aprillia and Syukri Arief + Open abstract View article PDF OPEN ACCESS A. H. Alkaff, H. Hendri, I. H. Farozy, M. Annisa and R.P. Aritonang + Open abstract OPEN ACCESS Characteristics of Alcian-blue Dye Adsorption of Natural Biofilm Matrix A Kurniawan, T Yamamoto, Sukandar and Guntur + Open abstract View article PDF
+ Open abstract Image: PDF OPEN ACCESS 012 Use of <i>Eichhornia crassipes</i> modified Nano-chitosan as a biosorbent for lead (II), cadmium (II), and copper (II) ion removal from aqueous solutions 012 A. H. Alkaff, H. Hendri, I. H. Farozy, M. Annisa and R.P. Aritonang + + Open abstract Image: PDF OPEN ACCESS 012 Characteristics of Alcian-blue Dye Adsorption of Natural Biofilm Matrix 012 A Kurniawan, T Yamamoto, Sukandar and Guntur —
OPEN ACCESS 012 Use of <i>Eichhornia crassipes</i> modified Nano-chitosan as a biosorbent for lead (II), cadmium (II), and copper (II) ion removal from aqueous solutions 012 A. H. Alkaff, H. Hendri, I. H. Farozy, M. Annisa and R.P. Aritonang + + Open abstract Image: PDF OPEN ACCESS 012 Characteristics of Alcian-blue Dye Adsorption of Natural Biofilm Matrix 012 A Kurniawan, T Yamamoto, Sukandar and Guntur 012
Use of <i>Eichhornia crassipes</i> modified Nano-chitosan as a biosorbent for lead (II), cadmium (II), and copper (II) ion removal from aqueous solutions A. H. Alkaff, H. Hendri, I. H. Farozy, M. Annisa and R.P. Aritonang + Open abstract IV View article PDF OPEN ACCESS Characteristics of Alcian-blue Dye Adsorption of Natural Biofilm Matrix A Kurniawan, T Yamamoto, Sukandar and Guntur
aqueous solutions A. H. Alkaff, H. Hendri, I. H. Farozy, M. Annisa and R.P. Aritonang + Open abstract View article PDF OPEN ACCESS O12 Characteristics of Alcian-blue Dye Adsorption of Natural Biofilm Matrix A Kurniawan, T Yamamoto, Sukandar and Guntur
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Determination of Parabens by Injection-Port Derivatization Coupled With Gas-Chromatography-Mass Spectrometry and Matrix Solid Phase Dispersion
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	pectrophotometric determination of cobalt in horse urine using 2-(5-bromo-2-pyridylazo)-5-[N-n-propyl-N-(3-sulfopropyl)amino]aniline as nromogenic reagent				
Arfidyaninggar Sep	- otia Rinda, Kanchana I	Jraisin, Akhmad Sabarudin, Duangjai Nacapricha and Prapin Wilairat			
+ Open abstract	View article	🔁 PDF			
OPEN ACCESS			012007		
The use of What	man-31ET paper for	r an efficient method for radiochemical purity test of ¹³¹ I-Hippuran			
Amal Rezka Putra,	Maskur, Yono Sugiha	rto, Chairuman, Adang Hardi Gunawan and Rohadi Awaludin			
	View article	PDF			
OPEN ACCESS			012008		
The Effect of Ter	nperature and Conc	centration of Foaming Agent to the β -Carotene Content in Product Derived from Carrots			
Qonitah Fardiyah, I	Barlah Rumhayati and	Yuniesti Husnul Khotimah			
	View article	PDF			
OPEN ACCESS Method Develop	ment for Pesticide l	Residue Analysis in Farmland Soil using High Perfomance Liquid Chromatography	012009		
1		barudin and Hermin Sulistyarti			
+ Open abstract	View article	PDF			
OPEN ACCESS			012010		
Molecularly Imp	rinted Polymers Ch	itosan-Glutaraldehyde for Monosodium Glutamate			
Ani Mulyasuryani,	Edi Haryanto, Hermir	Sulistyarti and Barlah Rumhayati			
	View article	PDF			
OPEN ACCESS			012011		
The Developmen	nt of Analytical Met	hod for the Determination of Azelaic Acid Content in Cosmetic Cream Products			
EHisisiaentişes Woiddo	ieso BrydcSofHindiiyngnto u	se this site you agree to our use of cookies. To find out more, see our Privacy and Cookies policy.	8		

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012012

Evaluation of Heavy Metal Exposure to Soil and Paddy Plant around the Closed Municipal Solid Waste Landfill: Case Study at Gunung Tugel Landfill, Banyumas-Central Java

Kasam, Suphia Rahmawati, Fajri Mulya Iresha, Dhandhun Wacano, Ida Farida Fauziah and Muhammad Afif Amrullah

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BIOCHEMISTRY

OPEN ACCESS	012013
Recombinant Protein Production from TPO Gen Cloning and Expression for Early Detection of Autoimmune Thyroid Diseases	
Aulanni'am Aulanni'am, Dyah Kinasih Wuragil, Djoko Wahono Soeatmadji, Zulkarnain and Agung Pramana W. Marhendra	
+ Open abstract View article PDF	
OPEN ACCESS	012014
Identification and Characterization of Bioactive Peptides of Fermented Goat Milk as a Sources of Antioxidant as a Therapeutic Natural Product	
Chanif Mahdi, Handayu Untari and Masdiana Cendrakasih Padaga	
+ Open abstract View article PDF	
OPEN ACCESS	012015
Green Chemistry Glucose Biosensor Development using Etlingera elatior Extract	
A Fatoni, M D Anggraeni, Zusfahair and H Iqlima	
+ Open abstract	

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Stability Test of Partially Purified Bromelain from Pineapple (Ananas comosus (L.) Merr) Core Extract in Artificial Stomach Fluid

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OPEN ACCESS			012017
In Vivo antiplate merr.)	let activity aggregat	ion assay of bromelain fractionate by ethanol from extract pineapple core (Ananas comosus [l.]	
F F Musfiroh, S Se	tiasih, S Handayani, S	Hudiyono and N M Ilyas	
+ Open abstract	View article	PDF	
OPEN ACCESS			012018
The Potency of E Ulcer Model Ind		Sauropus androgynus (L.) Merr Leaves as Therapeutic herbal of Rats (Rattus norvegicus) Peptic	
A Roosdiana, S A Y	/udandi and A Erika		
+ Open abstract	View article	PDF	
OPEN ACCESS			012019
• •	nesis of Glyserol-Co <i>rugosa</i> Lipase EC 3.	conut Oil Fatty Acid and Glycerol-Decanoic Acis Ester as Emulsifier and Antimicrobial Agents 1.1.3	
Sri Handayani, Ayu	ı Tanissa Tamara Putri,	Siswati Setiasih and Sumi Hudiyono	
+ Open abstract	View article	PDF	
OPEN ACCESS			012020
Depuration Study Extraction Metho		ead (Pb) and Copper (Cu) in Green Mussels Perna viridis through Continues-discontinues and Acid	
Budiawan, Ridla B	akri, Intan Cahaya Da	ni, Sri Handayani, Rizki Ade Kurnia Putri and Riska Tamala	
+ Open abstract	View article	PDF	
OPEN ACCESS			012021
The Proteomic A	nalysis of Pancreati	c Exocrine Insufficiency Protein Marker in Type 2 Diabetes Mellitus Patients	

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OPEN ACCESS			012022
The Effects of Ro	oot Extract Ruellia ti	uberosa L on Histopathology and Malondialdehyde Levels on the Liver of Diabetic Rats	
Alfin Nur Laily Ku	miawati, Aulanni'am, A	rie Srihardyastutie and Anna Safitri	
+ Open abstract	View article	PDF	
OPEN ACCESS			012023
Biosorption of Cr Trichoderma viria	· · ·	lution using Microorganisms: Comparison of the Use of Rhizopus oryzae, Bacillus firmus, and	
Anna Safitri, Putri I	Mahardini, Sasangka Pi	rasetyawan and Anna Roosdiana	
+ Open abstract	Tiew article	PDF	
OPEN ACCESS			012024
In vitro study of	DNA Adduct 8-OHd	G Formation by using Bisphenol A in Calf Thymus DNA and 2'-Deoxyguanosine	
Budiawan, Intan Ca	haya Dani, Ridla Bakri	i, Sri Handayani and Evi Ratna Dewi	
+ Open abstract	Tiew article	PDF	
OPEN ACCESS			012025
Fucoidan cytotox	icity against human	breast cancer T47D cell line increases with higher level of sulfate ester group	
Endang Saepudin, H	ʻildzah Alfita Qosthalar	ni and Ellya Sinurat	
	View article	PDF	
OPEN ACCESS			012026
Glucanase and Cl	nitinase from Some	Isolates of Endophytic Fungus Trichoderma spp.	
Sasangka Prasetyaw	van, Lilik Sulistyowati	and Aulanni'am	
	Tiew article	PDF	
This site uses cooki	es. By continuing to us	e this site you agree to our use of cookies. To find out more, see our Privacy and Cookies policy.	8

OPEN ACCESS			012027
Depigmentation	and Characterizatio	n of Fucoidan from Brown Seaweed Sargassum binderi Sonder	
Endang Saepudin,	Ellya Sinurat and Ira A	zmi Suryabrata	
	View article	PDF	
INORGANIC CI	HEMISTRY		
OPEN ACCESS			012028
Synthesis and Cl	haracterization of H	ydroxyapatite-Collagen-Chitosan (HA/Col/Chi) Composite Coated on Ti6Al4V	
Charlena, Ahmad H	Bikharudin and Setyant	to Tri Wahyudi	
	Tiew article	PDF	
OPEN ACCESS			012029
Molecular Self-A	Assembly of Group	11 Pyrazolate Complexes as Phosphorescent Chemosensors for Detection of Benzene	
N F Ghazalli, L Yu	liati and H O Lintang		
	Tiew article	PDF	
OPEN ACCESS			012030
Tempe Waste Wa	ater Degradation Us	ing TiO ₂ -N/Bentonite alginate Granule Photocatalyst with Ultraviolet Light Irradiation	
Aldila Khoirun Nis	saa', Sri Wardhani, Dan	ar Purwonugroho and Darjito	
	View article	PDF	
OPEN ACCESS			012031
Mg ²⁺ Doped into	o Electro-synthesize	d HKUST-1 and Their Initial Hydrogen Sorption Properties	
W W Lestari, D Ni	'maturrohmah, U S F A	Arrozi and H Suwarno	
	View article	PDF	

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Coordination Pol	ymer of M(II)-Pyra	zinamide (M = Co, Cd) with Double End-to-End Thiocyanate Bridge	
Yuniar Ponco Prana	anto		
	View article	PDF	
OPEN ACCESS	anosit From Bambo	o Fiber, Zeolite and Epoxy for Room Separation	012033
•		iki, Bangun Wasito and Suroso	
+ Open abstract	View article	PDF	
OPEN ACCESS Low Cost CaTiO	Pa Perovskite Synthe	esized from Scallop (<i>Anadara granosa</i>) Shell as Antibacterial Ceramic Material	012034
	ur Ilahi and Rismayan		
	View article	PDF	
OPEN ACCESS Synthesis of Hyd	lroxyapatite using P	recipitated Calcium Carbonate (PCC) from Limestones	012035
Sri Wardhani, Noor	· Isnaini Azkiya and R	achmat Triandi Tjahjanto	
✤ Open abstract	View article	PDF	
OPEN ACCESS			012036
Effect of metal ca	ation ratio on chem	ical properties of ZnFe ₂ O ₄ /AC composite and adsorption of organic contaminant	
Demara Meilia, Mo	ochamad Misbah Khur	ur and Tutik Setianingsih	
+ Open abstract	View article	PDF	
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Solmaz Aslanzadeh, Rahmat A. Kemal and Amadeus Y. Pribowo

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OPEN ACCESS			012038
Determination of pro	cess parameters	s for curcumin – dextrose cocrystallization	
Katherine, Denny Nugro	oho and Asaf K Su	ıgih	
+ Open abstract	View article	PDF	
OPEN ACCESS Synthesis and Charac	terization of Hy	ydroxyapatite Powder by Wet Precipitation Method	012039
S. E Cahyaningrum, N.	•		
+ Open abstract	View article	PDF	
OPEN ACCESS			012040
Effect of Evaporation	n Time on Separ	ration Performance of Polysulfone/Cellulose Acetate (PSF/CA) Membrane	
Intan Syahbanu, Bamba	ng Piluharto, Syał	urul Khairi and Sudarko	
+ Open abstract	View article	PDF	
OPEN ACCESS		a la la la Silia Thia Lassa Davies d'Essa Madadais al servitore (MTMS)	012041
-	•	ydrophobic Silica Thin Layer Derived from Methyltrimethoxysilane (MTMS)	
Adi Darmawan, Riyadir	ii Utari, Riza Eka	Saputra, Suhartana and Yayuk Astuti	
✤ Open abstract	View article	PDF	
OPEN ACCESS			012042
Micro Structure and	Hardness Analy	sis of Brass Metal Welded	
N Lukman Faris, Muljao	li and Djuhana		
	View article by continuing to us	PDF se this site you agree to our use of cookies. To find out more, see our Privacy and Cookies policy.	0

OPEN ACCESS			012043
The characteriza	tion of ceramic alun	nina prepared by using additive glass beads	
Suprapedi, Muljadi	i and Priyo Sardjono		
+ Open abstract	Tiew article	PDF	
OPEN ACCESS			012044
Effect of Heating	g Time to Density, H	lardness, and Resistivity Againt Fungus of Yellow Bamboo (Bambusa Vulgaris Var Schard. Vitata)	
A.H. Dawam Abdu	ıllah and Saukani Nasu	tion	
	Tiew article	PDF	
OPEN ACCESS			012045
Effects of Sodium	m and Chlorine Dop	ing on Optical Properties of Germanene: Density Functional Theory Calculation	
Mauludi Ariesto Pa	amungkas, Istiroyah, M	luhammad Fikri Salim and Dina Nurul Afifah	
	View article	PDF	
OPEN ACCESS			012046
Evaluation of the	e suitability of tin sla	ag in cementitious materials: Mechanical properties and Leaching behaviour	
Andi Rustandi, Fua	ad Wafa' Nawawi, Yudl	na Pratesa and Agung Cahyadi	
+ Open abstract	Tiew article	PDF	
OPEN ACCESS			012047
Corrosion Behav	vior of Carbon Steel	in Concrete Material Composed of Tin Slag Waste in Aqueous Chloride Solution	
Andi Rustandi, Agu	ung Cahyadi, Sonia Ta	ruli Siallagan, Fuad Wafa' Nawawi and Yudha Pratesa	
	View article	PDF	
OPEN ACCESS			012048
Characteristic of	Low Temperature (Carburized Austenitic Stainless Steel	
Tshiroyith, une sac Pak	iangRasçorSaroja Mu	Shuthionsiand Auror and Cookies and Shuthions and Cookies policy.	8

OPEN ACCESS			012049
The Effect of Var	rious Acids to the G	elation Process to the Silica Gel Characteristic Using Organic Silica	
NA Rahman, W Wi	diyastuti, D Sigit, M A	ajiza and W Sujana	
+ Open abstract	View article	PDF	
OPEN ACCESS			012050
Enhancing the Ha	ardness of Sintered	SS 17-4PH Using Nitriding Process for Bracket Orthodontic Application	
B Suharno, S Supria	adi, S T Ayuningtyas, '	Γ Widjaya and E R Baek	
+ Open abstract	View article	PDF	
OPEN ACCESS Effect of Shot Per 316L Biomaterial	•	Shot Distance and Shot Angle on Surface Morphology, Surface Roughness and Surface Hardness of	012051
Erich Umbu Kondi	Maliwemu, Viktor Ma	alau and Priyo Tri Iswanto	
+ Open abstract	View article	PDF	
OPEN ACCESS Synthesis and Ch	aracterization Pecti	n-Carboxymethyl Chitosan crosslinked PEGDE as biosorbent of Pb(II) ion	012052
•	Siswanta, Mudasir and	• •	
+ Open abstract	View article	PDF	
OPEN ACCESS The Use of Electr	rochemical Impeda	nce Spectroscopy Method for Corrosion Resistance Evaluation of 2205 and 410s Stainless Steels in	012053
	Chloride Solution		
Andi Rustandi, Ger	aldo Sirait, Andy Sapu	atra and Yunita Sadeli	
The see the tractori	es. By VonWnthing to u	se this site you agree to our use of cookies. To find out more, see our Privacy and Cookies policy.	•

OPEN ACCESS			012054
Characterization Method	Studies of Cyclotro	n CS-30 Carbon Puller Material Using Powder X-Ray Diffraction and SEM, EDX Cross Section	
S Febriana, E S Riy	vanto, A Dimyati and A	Handayani	
	View article	PDF	
OPEN ACCESS			012055
Preparation and (Characterization of 1	Dabco (1,4-Diazabicyclo [2.2.2]octane) modified bentonite: Application for Congo red removal	
Tarmizi Taher, Ded	i Rohendi, Risfidian M	Iohadi and Aldes Lesbani	
+ Open abstract	View article	PDF	
OPEN ACCESS			012056
Study on the curi	e transition of P(VI	DF-TrFE) copolymer	
Devi Eka Septiyani	Arifin and J J Ruan		
✤ Open abstract	View article	PDF	
OPEN ACCESS			012057
Synthesis carbon	foams prepared fro	m gelatin (CFG) for cadmium ion adsorption	
M. Ulfa and D.K. U	Jlfa		
+ Open abstract	View article	PDF	
MOLECULAR S	CIENCE AND COM	APUTATIONAL CHEMISTRY	
OPEN ACCESS			012058
Theoretical Study	y of Electron Transf	Fer Properties of Squaraine Dyes for Dye Sensitized Solar Cell	
Ratna Juwita and H	ui-Hsu Gavin Tsai		
+ Open abstract	View article	PDF	
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OPEN ACCESS			012059
•	-	toire of siRNA Linc-ROR and siRNA mRNA ARF6 from the lincRNA-RoR/miR-145/ARF6 the progression of Triple Negative Breast Cancer	
Arli Aditya Parikes	it and Rizki Nurdiansy	ah	
	View article	PDF	
OPEN ACCESS			012060
<i>Ab-initio</i> study or relaxation-effect		ctrum of color change sapphire based on first-principles calculations with considering lattice	
Mega Novita, Hika	ri Nagoshi, Akiho Sud	o and Kazuyoshi Ogasawara	
+ Open abstract	Tiew article	PDF	
OPEN ACCESS Effects of alkano I Surya, S Maulina	-	tion in natural rubber and epoxidized natural rubbers compounds	012061
+ Open abstract	View article	PDF	
NANOMATERIA	ALS AND NANO DE	VICES	
OPEN ACCESS			012062
Synthesis and Ch Crosslinker	naracterization of Fe	₃ O ₄ Nanoparticles using Polyvinyl Alcohol (PVA) as Capping Agent and Glutaraldehyde (GA) as	
Lale Budi Hutami I	Rahayu, Ika Oktavia W	ulandari, Djoko Herry Santjojo and Akhmad Sabarudin	
+ Open abstract	View article	PDF	
OPEN ACCESS			012063
The Role of Mod	lification SBA-15 M	lesoporous Silica with CPTMS in Cd Adsorptions	
Donanta Dhaneswa	ra, Jaka Fajar Fatrians	yah, Aloysius Brahmarsi Mahagnyana, Farhan Delayori, Dewanto Adhy Putranto and Siti Utami Agustina Adriyan	i Anwar
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OPEN ACCESS Preparation and (Characterization of (Chitosan-coated Fe ₃ O ₄ Nanoparticles using Ex-Situ Co-Precipitation Method and	012064	
Tripolyphosphate/Sulphate as Dual Crosslinkers				
Ika O Wulandari, V	vita T Mardila, D J Djo	ko H Santjojo and Akhmad Sabarudin		
	View article	PDF		
OPEN ACCESS			012065	
Ex-Situ Synthesi	s of Polyvinyl alcoh	ol(PVA)-coated Fe ₃ O ₄ Nanoparticles by Coprecipitation-Ultrasonication Method		
Imam Riva'i, Ika O	ktavia Wulandari, Herr	nin Sulistyarti and Akhmad Sabarudin		
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Insertion of bentonite with Organometallic [Fe₃O(OOC₆H₅)₆(H₂O)₃(NO₃).nH₂O] as Adsorbent of Congo Red

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Abstract: The adsorption of Congo red using bentonite inserted organometallic has been investigated. The insertion bentonite was characterized using FT-IR Spectrophotometer, XRD and XRF analysis. The FT-IR characterization showed the higher intensity of peak wavenumber at 470.6 cm⁻¹ for Fe₃O on the ratio 1:3. While the XRD characterization showed the shift of diffraction angle of 2θ was 5.2° and has a basal spacing of 16.8 Å. In the XRF characterization, the insertion process of organometallic occurred optimally with the percentage of metal oxide reached 71.75 %. The adsorption process of bentonite inserted organometallic compound [Fe₃O(OOC₆H₅)₆(H₂O)₃(NO₃)·nH₂O] showed the adsorption rate (k) is 0.050 min⁻¹, the largest adsorption capacity (b) at 70°C is 4.48 mol/g, the largest adsorption energy at temperature 30°C which is 6.4 kJ/mol Organometallic compounds. The value of the enthalpy (Δ H) and entropy (Δ S) decreased with increasing concentrations of the Congo red. Effect of pH on the adsorption on at pH 3 shows the biggest of number Congo red absorbed is 19.52 mg/L for insertion of bentonite.

Keywords: Bentonite, [Fe₃O(OOC₆H₅)₆(H₂O)₃(NO₃)·nH₂O], Congo red dye

1. Introduction

Organics pollutant such as coloured dye can be found industries effluent such as textiles, rubber, paper, plastic, cosmetics, etc. Discharging dyes into river although in a small amount can affect the organisme life. As dyes are designed resist with time and exposure to sunlight, they cannot be easily removed by conventional wastewater treatment. Thus, dye removal has been an important but challenging area of wastewater treatment. To remove dyes and other colored contaminants, the famous method used was adsoption [1].

Bentonite is a layered material or clay containing inorganic minerals found in nature [2]. Bentonite is used as an adsorbent [3]. Bentonite in its application as an adsorbent has deficiency among others containing impurities minerals so that its absorption is not optimal. Therefore, to optimize the absorption of bentonite activation needs to be done. The activation process aims to separate impurities naturally on bentonite. In the activation process, there are 2 ways done that are by physical way or chemical way.

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In the physical process, combustion at high temperatures causes the removal of water molecules from the crystal sequence, so that two adjacent OH groups release one molecule of water [4], while in the chemical process using sulfuric acid aims to remove the metals of size small. This is due to the metals present in bentonite bound to the H $^+$ ions derived from the acid thus the layer acting as the insertion admission will be more open. Bentonite modification is expected to produce bentonite which has high adsorption ability and can perform insertion process that produces insulated atomic bentonite, molecule or complex compound.

Said and Palapa[5] found that the insertion process using Al_2O_3 metal oxide shows a slight increase in the distance between layers. This is indicated by the variation of contact time of adsorption of bentonite inserted metal oxide compound Al_2O_3 to ammonia at 4, 8, 16, 24 hours resulted that at 24 hours bentonite inserted Al_2O_3 able to adsorb ammonia with the largest percentage, while in an adsorption process, time 24 hours is so long that bentonite inserted Al_2O_3 metal oxide compounds can be said not yet optimally used as an adsorbent because of the size of the cation possessed by small metal Al_2O_3 oxide to become inserted bentonite.

Therefore, in this research, bentonite insertion process using organometallic compound as insertion will be done. The organometallic compounds $[Fe_3O(OOC_6H_5)_6(H_2O)_3(NO_3)\cdot nH_2O]$ have advantages such as high reactivity properties and larger cation size (macrocation). It is expected that bentonite insertion of organometallic compounds capable of inserting optimally and able to increase high adsorption capacity.

Bentonite inserted macrocation of organometallic compounds characterized using FT-IR, XRD, and XRF spectrophotometers to see the metal oxide composition. Further, bentonite inserted an organometallic compound to be used as an adsorbent to adsorb the Congo red dye. In this study, the selected adsorbate is Congo red dye, because this dye includes hazardous waste. The Congo red adsorption process that occurs can be affected by several conditions including the influence of adsorption time, the influence of concentration and temperature, and the pH of the solution. Determination of equilibrium concentration on the adsorption of activated bentonite and bentonite insertion of the organometallic compound was analyzed using UV-Vis spectrophotometer.

2. Materials and Method

2.1. Material

The materials used in this study are natural bentonite, Congo red dye, sulfuric acid, aquadest, iron (III) nitrate nonahydrate [Fe (NO₃)₃.9H₂O], benzoic acid, Sodium hydroxide 0.1 M, and hydroxide acid 0.1 M. The characterization of bentonite was done with X-Ray spectroscopy Diffractometer (Shimadzu lab X-type 6000), FT-IR spectrophotometer (Shimadzu-Prestige-21), X-Ray fluorescence, PAN Analytical Type Minipod 4) and Uv-Vis spectrophotometer (Thermo Scientific Geneysis 20).

2.2. Bentonite inserted with Organometallic Compounds $[Fe_3O(OOC_6H_5)_6(H_2O)_3(NO_3) \cdot nH_2O]$

A total of 5 g of bentonite was dissolved in 200 mL of aquadest. A 15 g of the organometallic compound acting as the insertion compound is prepared by adding 100 mL of 1 M NaOH, then mixed in reflux and stirred vigorously for 24 hours at room temperature for 1 day while being fed by nitrogen gas. The resultant insertion of organometallic compounds is then washed with aquades and dried in open-air for 24 hours. The results of bentonite insertion of organometallic compounds were characterized using XRD, FTIR, and XRF spectroscopy.

2.3. Study of Adsorption of Congo Red

2.3.1. Effect of adsorption time

A total of 0.03 grams of bentonite adsorbent inserted organometallic compound and activated bentonite (control) were added to 50 mL Congo red with a concentration of 150 mg / L then stirred with a horizontal shaker at 30 min intervals. Variations in absorption time varied from 10, 20, 30, 40, 50, 60, 70, 80, and 90 min. Congo red which has gone through the adsorption process is silenced then measured its concentration by using Uv-Vis spectrophotometer to find out

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2.3.2. Effect of Concentration and Temperature

The thermodynamic studies of dye adsorption on bentonite inserted macrocation adsorbents were carried out through a series of experiments by varying the concentration of dyestuff and adsorption temperature. A total of 0.03 g of adsorbent was mixed with 50 mL of 25, 50, 75, 100 and 150 (mg/L) dye solutions used as controls and for bentonite insertion of its organometallic compound using dye solutions of 10, 20, 30, 40 and 50 (mg/L), then stirred using a horizontal shaker for 1 hour at varying temperatures (30, 40, 50, 60, and 70°C). The solution was silenced and measured using a UV-Vis spectrophotometer to determine the residual concentration and dyestuff adsorbed concentration after adsorption.

2.3.3. Effect of pH

The effect of pH on dye adsorption on bentonite inserted organometallic compounds was studied by varying the initial pH of the dye solution. Adsorbent bentonite inserted as much as 0.03 grams was added to 50 mL dye solution at 25 mg / L concentration and then stirred with a horizontal shaker at room temperature for one hour. The initial pH of the dye solution is set at 3, 4, 5, 6, 8, 9, 10, 11, and 12 for inserted bentonite while for the activated bentonite (control) the initial pH of the dye solution is set to 1, 2, 3, 4, 5, 6, 8, 9, 10, 11, and 12 were added with 0.1 M NaOH addition and 0.1 M HCl of solution was allowed to stand for 10 min and measured using UV-Vis spectrophotometer to determine residual concentration and adsorbed concentration of the substance Color after the adsorption process.

2.3.4. Congo red Interaction Studies

The interaction between the adsorbent and the adsorbate is studied spectroscopically by looking at changes occurring in the adsorbent before and after the adsorption process. As much as 0.03 g of adsorbent bentonite insertion of the organometallic compound was mixed with 50 mL of dye with concentration 50 mg/L. The mixture is stirred using a horizontal shaker for 1 hour, then the mixture is separated through the filtration process. The adsorbents are then dried with an oven at 80°C. Adsorbents that have been used for the adsorption process are then analyzed using FT-IR to determine the functional group changes occurring before and after the adsorption process.

2.4. Data Analysis

The adsorption process is studied through kinetic and thermodynamic parameters. The adsorption kinetic is studied by variation of adsorption time and adsorption rate calculated based on Langmuir-Hinnshelwood adsorption equation as follows:

$$\frac{\ln(\frac{c_0}{c})}{c} = k_1 \frac{t}{c} + K \tag{1}$$

where:

- *C*₀ = the initial concentrations
- K_1 = the rate of adsorption
- C = Concentrate Congo red after time
- t = Adsorption time
- K = Adsorption equilibrium constant

The thermodynamic parameters studied through variations in the concentration of Congo red dye, adsorption capacity and adsorption energy were calculated on the basis of the Langmuir equation as follows:

$$\frac{C}{m} = \frac{1}{bK} + \frac{C}{b} \tag{2}$$

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$$E = -RT \ln K$$
(3)

where:

- C = The concentration of Congo red after adsorption reaches equilibrium
- m = Mol Congo red adsorbed
- K = Equilibrium constant
- b = Adsorption Capacity
- E = Adsorption Energy
- R = Constanta

T = Temperature

While to find the value of coefficient of adsorbate distribution used equation as follows:

$$\ln \mathrm{Kd} = \frac{\Delta S}{\mathrm{R}} - \frac{\Delta \mathrm{H}}{\mathrm{R}T} \tag{4}$$

.where:

Kd = Coefficient of adsorbate distribution (qe/Ce)

 $\Delta H = Entalphi$

 $\Delta S = Entrophi$

R = Constanta

T = Temperature

3. Results and Discussion

3.1. Effect of Adsorption Time

The adsorption time calculation data on the amount of adsorbed Congo red can be seen in Figure 1.

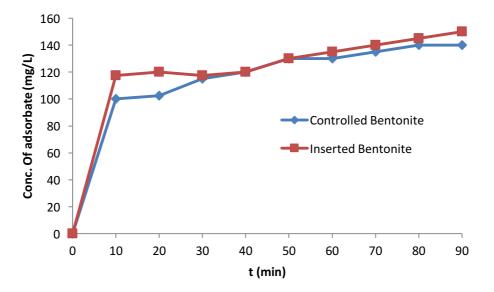


Figure 1. Influence of contact time of activated bentonite (control) and inserted bentonite to the amount of adsorbed Congo red

Figure 1 shows as the adsorption time increase, the greater the concentration of the adsorbed Congo red dye. In bentonite inserted to see the amount of the adsorbed red dye Congo increase within 20 minutes as it reaches the equilibrium time. This shows that bentonite inserted organometallic

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compound has reached the adsorption equilibrium. Adsorption time data can be used to determine the kinetic parameters calculation adsorption. Data of kinetic parameters presented in Figure 2 and b.

Based on the Langmuir-Hinshelwood equation, the adsorption constant rate can be calculated as the slope of the ln (Co/C)/C plot to t/C as the line equation. The value of each slope as the rate of adsorption constant (k1) and the correlation coefficient (R^2) of the plot (Co/C)/C to t/C is presented in Table1. The correlation coefficient obtained shows linear line equation. The equation of the line with the correlation coefficient (> 0.9) shows that the Congo adsorption kinetics credits the Langmuir-Hinshelwoodoold kinetics equation.

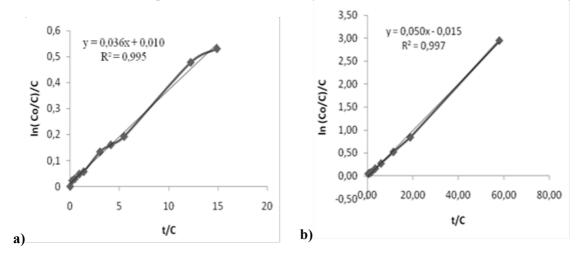


Figure 2. The bentonite adsorption kinetic parameters: a) controlled, and b) inserted bentonite

Table 1. Values of adsorption rates for Activated Bentonite (control) and Inserted Bentonite

Adsorbent	Rate of adsorption (min ⁻¹)	\mathbf{R}^2
Activateded bentonite	0.036	0.995
Inserted bentonite	0.050	0.997

It is shown that the activated bentonite have the good ability as adsorbents. This is based on the basal spacing between the activation and inserted bentonite. From the previous study [6], It is found that the activated bentonite at 2 per angle shift has basal spacing 24 Å while inserted bentonite has basal spacing of 16,8 Å. The difference of basal spacing has only a slight gap. The more differences between them happened because of the adsorbate enter to inserted bentonite layer by layer. Solute attaches to the surface of montmorillonite and diffuses into the pores before finally being trapped inside of the adsorbent. The solute will be continuously deposited on the surface of the adsorbent and the layer will become thicker. The interaction force, between the solute and the adsorbent, means that the solutes cannot leave the adsorbent [7]. It is also known that the montmorillonite crystal structure consists of three layers: two units of the tetrahedral layer and one octahedral layer [8]. It can be said that the inserted bentonite rate of adsorption is higher than the activated one.

3.2. Effect of Concentration and Temperature

The curve of adsorption as the effect of concentration and temperature on the amount of adsorbed Congo red was presented in Figure 3a and b. The figures show the influence of temperature and concentration on the adsoprtion process of Congo red. It can be seen from the figure that the activated bentonite show the better performance than the inserted bentonite with it marked by the higher value of adsorbate concentration. This phenomenon was explained by another researcher[9,10]. The Congo red color has a S = O vibration with a very low intensity at 1072 cm⁻¹ wave numbers. In addition, the aromatic C = C vibration appears at 1481 cm⁻¹ wave numbers. The symmetric stretching vibration of the organometallic compound will reduced before the adsorption[9]. In addition, the adsorption capacity decreased with the increasing concentration. It is found that the higher temperature is to the

advantage of adsorption and that the adsorption is an endothermic reaction. It is well known that increasing temperature may produce a swelling effect within the internal structure of absorbent, penetrating the large dye molecule also will block the pore of adsorbent. Finally, it will reduce the adsorbent performance[10].

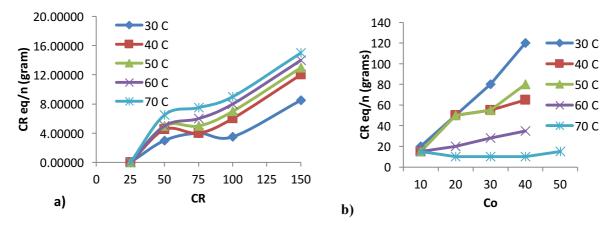


Figure 3. Effect of adsorption temperature and concentration: a) activated, and b) inserted bentonite

In addition, Figure 3 shows the number of Congo red adsorbed by activated bentonite greater than the inserted bentonite. This is due to the activated bentonite has a spacing between layers very small so when the adsorbate, Congo red directly entry to the inside of adsorbent, it is more easily filled compared to the inserted bentonite that has a greater spacing between layers. It can be concluded the activated bentonite is more reactive than inserted bentonite [11]. Furthermore, we calculate the kinetic and thermodinamic parameters using the equation 2,3 and 4. The result of kinetic parameter such as adsoprtion capacity and energy of adsorption are list in Table 2 while the thermodynamic parameters such as changes in Gibbs (ΔG°), enthalpy (ΔH°) Entropy (ΔS°) are list in Table 3.

Table 2. The kinetic parameter of adsorption process of activated and inserted bentonite										
Adsorbent	Activated bentonite					Inserted bentonite				
Temp. (c)	30	40	50	60	70	30	40	50	60	70
b (mg/g)	15.6	43.4	71.4	62.5	58.8	0.35	0.59	0.72	1.33	4.48
E (kJ/mol)	-7.64	-5.32	2.21	7.02	10.9	-6.4	-3.83	-4.22	-1.93	-0.08

Table 2. The kinetic parameter of adsorption process of activated and inserted bentonite

Table 3. The thermodynamic parameter of adsorption process of activated and inserted bentonite

Adsorbent	Activated bentonite				Inserted bentonite					
Conc. (ppm)	25	50	75	100	150	10	20	30	40	50
$\Delta H (kJ/mol)$	69.5	81.8	59.1	54.6	42.2	59.4	153.2	190.2	198.3	124.2
ΔS (kJ/mol)	0.256	0.292	0.215	0.2	0.155	0.109	0.263	0.325	0.336	0.212

The adsorption capacity (b) of the activated bentonite (control) increased from 30 to 50° C but at 60 and 70° C, the adsorption capacity decreased. The inverted results was found to the inserted bentonite, the capacity of adsorption (b) has improved very well as temperature increases. This shows that in bentonite activated its optimum adsorption capacity at 50° C. For the adsorption energy, it is seen that the temperature of 70° C has the highest energy of adsorption. Further, the highest enthalphy and entropy occurs in inserted bentonite with Congo red concentration of 40 mg/L were 198.3 kJ/mol and

0.336 kJ/mol, respectively. This happens because adsorbent adsorbate irregularly occurs on the surface only and into the surface, it appears that even though the energy is large but the adsorption capacity is not so great and vice versa if the energy is small but it has a large amount of capacity.

3.3. Effect of pH

Effect of pH on the adsorption of Congo red dye is showed in Figure 4.

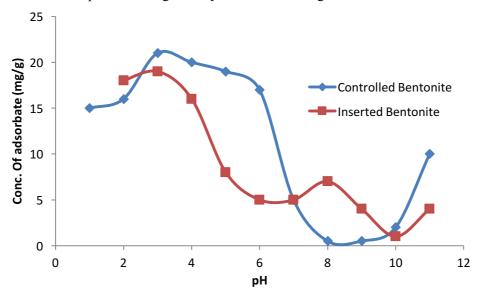


Figure 4. Effect of activated bentonite adsorption pH (control) and bentonite insertion of organometallic compound to the amount of Congo red adsorbed

The Congo red dye adsorption process by activated bentonite (control) has an initial pH of 4. After added the hydrochloric acid (HCl), the pH reached 3 and consequently the number of Congo red adsorbed was increased. In vice versa, after pH reahed 2 and 1, there was a decrease in the amount of adsorbed (Congo red). In the inverted process by adding process the sodium hydroxide (NaOH), the reducing number of Congo red was existed. However, in the of addition of sodium hydroxide (NaOH) so that the pH reaches 6 there is a decrease in the amount of adsorbed red Congo characterized by a concentration of Congo red dyestuff adsorbed by bentonite inserted into dark red. Two possible mechanisms of adsorption of Congo red may be considered: (a) electrostatic interaction between the protonated groups of bentonite and the dye, and (b) the chemical reaction between the adsorbate and the adsorbent [12].

As the pH was 8, the concentration of adsorbate adsorbed rises again. This is because of the more the addition of NaOH the more the amount of adsorbate is adsorbed and the solution is marked to be pink. Similarly, activated bentonite, Congo red adsorption by the adsorbent inserted bentonite organometallic compounds also showed a decrease in the amount of Congo red adsorbed at pH 8, 9, 10 followed by changes in the blend color to red tea. The existence of Congo red discoloration on adsorbed by the activated and inserted bentonite either in acidic or alkaline conditions based on pH of Congo red dye which naturally has effective pH values in the range 3-6. When the atmosphere is a mixture acid solution then changes color to purple, whereas under alkaline conditions the mixture becomes colorless Congo red.

3.4. Interaction Studies of Activated and Inserted Bentonite with Organometallic Compounds using FT-IR

The gradual changes occur in the vibrational frequency associated with a specific chemical bond. It concerning the peak position shift of IR spectra are based on the notion that the extent of frequency shift can be directly correlated with the level of specific molecular interactions, such as hydrogen

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bonding and dipole–dipole interactions. This also applies to the Congo red adsorption interaction study by activated and inserted adsorbent during before and after adsorption. Factors that affect the vibrational IR are acid activation, inserted organometallic and adsorption of congo red dye stuff process. During the acid activation process, the acid attack protons penetrate into the clay mineral layers and attack the structural OH groups[13]. By modification of bentonite through insertion of organo metallic will cause the basal spacing larger and consequently the adsorption capacity to increase. The existance of organometallic can be proven from shifting in IR spectra [14]. The vibration of the functional groups present in the Congo red dye will appear on the FT-IR spectrum with a typical wave number. The FTIR spectra before and after the activated bentonite adsorb Congo red dye contained was presented in Figure 5, while the spectra of inserted bentonite before and after Congo red dye adsorption was presented in Figure 6.

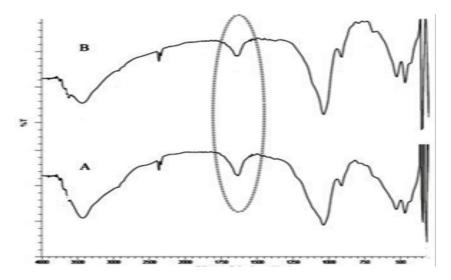


Figure 5. FTIR spectra of activated bentonite: a) before, and b) after adsorb the Congo red dye

Figure 5 shows the activated FT-IR bentonite spectrum changes before and after adsorb the noncontrasting Congo red substance. However, when viewed from the number of waves there is a difference of each bonding vibration. In the wave number 1041.5 cm⁻¹ shows the vibration of Si-O-Si strain on bentonite and S = O of the Congo red dye with high intensity and sharpness. In addition, the bonding vibrations of bentonite after adsorbing Congo red appearing at the wave number 1635 cm⁻¹ denotes the N=N vibration for the Congo red as a sign that the Congo red dye is an azo group.

Vibration NH contained in Congo red regional group appears at wave number 3618.4 cm⁻¹[15]. Figure 6 shows the change in the spectrum FT-IR bentonite inserts an organometallic compound before and after adsorb the visible Congo red dye. On the spectrum of inserted bentonite that have to adsorb dye Congo red visible vibration new bond indicates the group functional Congo red, which is the wave number 1635.6 cm⁻¹, which shows the absorption band for N=N as a marker of group azo.

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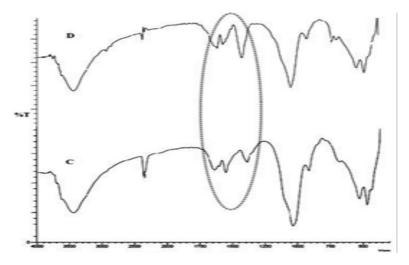


Figure 6. FTIR spectra of inserted bentonite: c) before, and d) after adsorb Congo red dye

4. Conclusion

The activated bentonite has smaller adsorption rate than the inserted bentonite i.e. 0.036 min⁻¹ and 0.050 min⁻¹, respectively. The adsorption capacity of activated bentonite is greater than the inserted bentonite adsorbent as temperature increases. The greatest adsorption energy of activated bentonite was 11 kJ/mol at 60°C, while inserted bentonite was 6.4 kJ/mol at 30°C. The enthalpy of activated and inserted bentonite decreased with increasing the Congo red concentration. The lowest entropy was occured at 50 mg/L by activated bentonite of 0.155 kJ/mol, whereas in the lowest entropy of inserted bentonite was occured 0.109 kJ/mol at Congo red concentration of 10 mg/L. At pH 3 there was an increase in the amount of Congo red adsorbed by activated bentonite and inserted bentonite i.e. 20.45 mg/L and 19.52 mg/L, respectively.

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