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SEMINAR NASIONAL

Perkembangan Riset dan Teknologi di Bidang Industri Ke-19



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di Bidang Industri Ke-19

**PANITIA SEMINAR NASIONAL
PERKEMBANGAN RISET DAN TEKNOLOGI
DI BIDANG INDUSTRI KE-19**

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Fakultas Teknik Universitas Gadjah Mada*

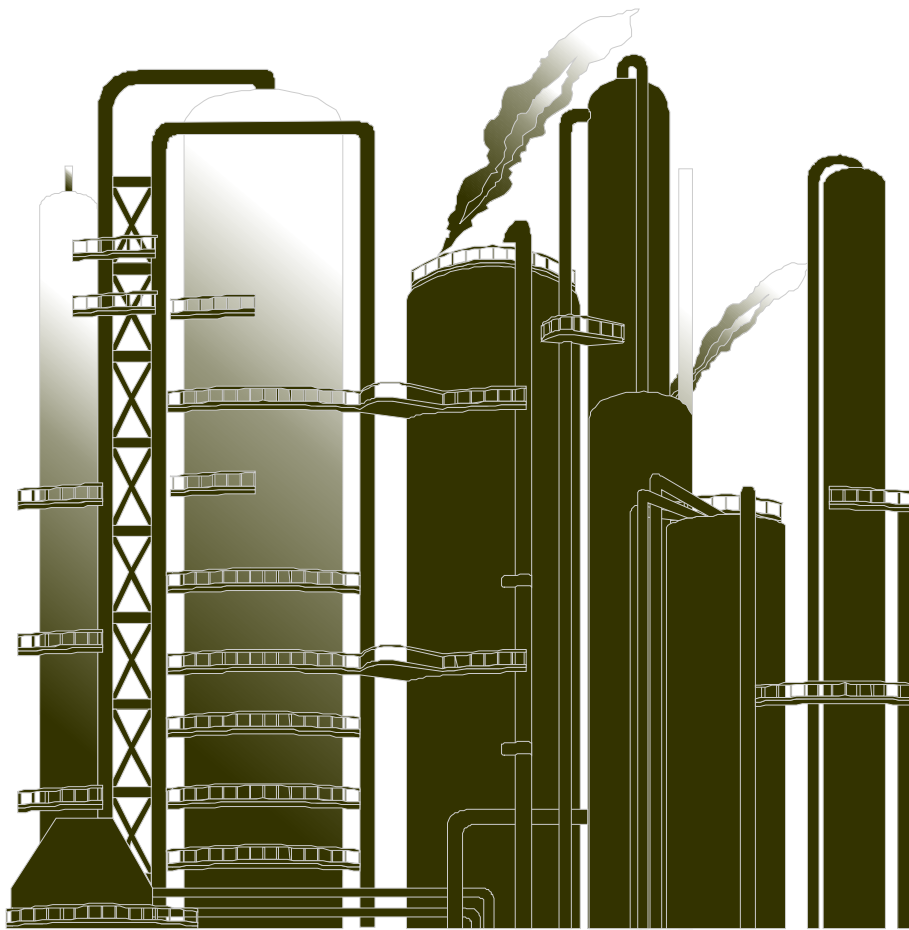
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Kantor Pusat Fakultas Teknik UGM
Yogyakarta, 4 Juni 2013



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**Prosiding Seminar Nasional
Perkembangan Riset dan Teknologi di Bidang Industri ke 19**

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KATA PENGANTAR

Seminar Nasional Perkembangan Riset dan Teknologi Di Bidang Industri yang ke 19 yang dilaksanakan tanggal 4 Juni 2013, bertempat di Kantor Pusat Fakultas Teknik UGM merupakan seminar rutin yang diselenggarakan oleh Pusat Studi Ilmu Teknik (PSIT) Universitas Gadjah Mada. Seminar ini terlaksana atas kerjasama antara PSIT UGM dengan Jurusan Teknik Mesin dan Industri, dan dengan Jurusan Teknik Kimia, Fakultas Teknik UGM. Seminar nasional ini merupakan forum diskusi dan tukar informasi bagi para peneliti, praktisi di bidang industri dan diharapkan dapat menghasilkan interaksi yang sinergis antara akademisi dan praktisi sehingga dapat mempercepat peningkatan laju perkembangan industri nasional.

Dalam seminar ini telah disampaikan 39 makalah yang terbagi dalam sub topik : Bahan Teknik dan Mekanika Bahan, Kendali Proses, Perpindahan Panas dan Massa, Teknik Reaksi dan Teknik Pembakaran, Mekanika Fluida, Pengolahan Limbah Industri dan Lingkungan, Teknik Industri.

Prosiding seminar ini diharapkan dapat memberikan informasi perkembangan yang paling mutakhir dalam bidang riset dan teknologi di bidang industri di Indonesia. Panitia telah berusaha semaksimal mungkin untuk menyusun semua makalah dalam bentuk prosiding yang representatif, namun masukan dan kritik dari para pembaca masih sangat diharapkan.

Seminar ini dapat terlaksana dengan sukses berkat partisipasi dan bantuan dari berbagai pihak. Panitia mengucapkan terima kasih kepada para pemakalah, para peserta, PT Global Haditech dan serta semua pihak yang telah membantu penyelenggaraan acara seminar.

Yogyakarta, 5 Juli 2013

**Panitia Seminar Nasional
Perkembangan Riset dan Teknologi Di Bidang Industri ke 19**

DAFTAR ISI

Kata Pengantar	iii
Daftar Isi	v

BAHAN TEKNIK - MEKANIKA BAHAN

1	Karakteristik Proses Hidriding-Dehidriding Pada Paduan U-7Mo <i>Agoeng Kadarjono, Supardjo</i>	BT/MB – 1
2	Perancangan Kekuatan Sirip Roket RX 320 Akibat Pengaruh Beban Aerodinamik yang Terjadi <i>Agus Budi Djatmiko</i>	BT/MB – 9
3	Perancangan Struktur Tabung Roket RX 450 LAPAN Akibat Pengaruh Getaran Yang Terjadi <i>Agus Budi Djatmiko</i>	BT/MB – 16
4	<i>Springback</i> Pada Pembentukan Plat Baja Dengan <i>Air Vee Bending</i> <i>Benidiktus Tulung Prayoga</i>	BT/MB – 24
5	Analisis Konsumsi Energi Listrik pada PC-Base CNC Dengan Variasi Parameter Pemesinan <i>Budi Basuki, Herian, Gesang Nugroho</i>	BT/MB – 30
6	Pengaruh Nitrokarburising Terhadap Kekerasan Baja AISI 410 <i>Edy Iryanto , Viktor Malau, Tjipto Sujitno</i>	BT/MB – 36
7	Analisis Pengaruh Penggunaan Sistem <i>Minimum Quantity Lubrication (MQL)</i> Dengan Variasi Fluida <i>Lubricant</i> Minyak Nabati Terhadap Keausan Pahat. <i>Istyawan Priyahapsara, Herianto, Herliansyah</i>	BT/MB – 42
8	Variasi Waktu Implantasi Ion Chrom untuk Meningkatkan Hardness dan Menurunkan Corrosion Rate dalam Media Phosphat Bayer Salin Material Penyambung Tulang <i>Priyo Tri Iswanto, Tjipto Sujitno , Bangun Pribadi</i>	BT/MB – 48
9	Pengaruh Perlakuan Panas T6 dengan Variasi Suhu <i>Aging</i> terhadap <i>Fatigue Crack Growth Rate</i> Paduan Aluminium A356 <i>Priyo Tri Iswanto, Muhammad Yusuf Kurniawan</i>	BT/MB – 54
10	Peningkatan Katahanan Korosi Material <i>Implant SS304</i> dalam Media <i>Phosphat Bayer Salin</i> dengan <i>Electroplating Ni-Cr</i> <i>Priyo Tri Iswanto, Soekrisno, Nani Mulyaningsih</i>	BT/MB – 61
11	Pengaruh Temperatur Anil terhadap Ketangguhan dan Ketahanan Korosi Paduan Fe-2,2Al-C <i>Ratna Kartikasari</i>	BT/MB – 68
12	Analisis Distribusi Tegangan Pada Struktur Nosel Motor Roket RX-450 Akibat Beban Tekanan dan Temperatur <i>Setiadi</i>	BT/MB – 74

- | | | |
|----|---|-------------|
| 13 | Karakterisasi Mekanis Lapisan Nickel, Hard Chrome dan Nickel-Hard Chrome pada Permukaan Baja AISI 410
<i>Viktor Malau, Soekrisno, Adityo Noor Setyo</i> | BT/MB – 80 |
| 14 | Studi Laju Korosi dan Kekerasan Baja AISI 1030 yang Diberi Lapisan Nickel Electroplating dengan Variasi Konsentrasi Larutan NaCl dan Lama Pelapisan
<i>Viktor Malau, Nelson Seleman Lupp</i> | BT/MB – 88 |
| 15 | Distribusi Mesh Serbuk Duralumin pada Penggilingan Bola Sebulung Putar D200 Putaran Cepat
<i>Wahyono Suprpto</i> | BT/MB – 95 |
| 16 | Pengembangan Chassis model Ladder Frame untuk Mobil Pick Up Niaga Multiguna
<i>Yusuf Kadang dan I Made Londen Batan</i> | BT/MB – 102 |

KENDALI PROSES

- | | | |
|---|--|--------|
| 1 | Kehilangan Vitamin C pada Buah dan Sayuran Selama Proses Pengeringan dan Pengolahan, Bagaimana Solusinya?
<i>Suharwadji Sentana</i> | KP – 1 |
|---|--|--------|

MEKANIKA FLUIDA

- | | | |
|---|--|---------|
| 1 | Pengaruh Flow Ratio Terhadap Pressure Ratio pada LJGP Dengan Area Ratio 0,65 dan Throat Ratio 4
<i>Dandung Rudy Hartana, Daru Sugati, Indarto, Purnomo, Eswanto</i> | MF – 1 |
| 2 | Perancangan Generator Magnet Permanen Flux Aksial untuk Aplikasi Turbin Angin Vertikal
<i>F. Eko Wismo Winarto, Sugiyanto</i> | MF – 6 |
| 3 | Optimasi Perletakan Kompresor Pada Jaringan Perpipaan Gas
<i>Joko Waluyo</i> | MF - 12 |
| 4 | Pengaruh Jarak Celah Annulus dan Tekanan Operasi terhadap Kinerja <i>Rotating Filter</i>
<i>Prajitno, Danni A.K.</i> | MF – 19 |

PENGOLAHAN LIMBAH INDUSTRI DAN LINGKUNGAN

- | | | |
|---|--|--------|
| 1 | Pelepasan Terkendali Dan Pengembangan Minyak Kayumanis Sebagai Pengawet Alami Pada Pengemas Aktif Berbasis Selulosa dan Evaluasinya Pada Ikan Lemuru (<i>Sardinella Longiceps</i>)
<i>Margaretha Tuti Susanti</i> | PL – 1 |
| 2 | Penurunan Kandungan Rhodamine B dalam Air Limbah Industri Tekstil dengan Proses Elektrokoagulasi dan Fotokatalitik
<i>Sutanto, Danang Widjajanto, dan Hidjan</i> | PL – 7 |

PERPINDAHAN KALOR DAN MASSA

- 1 Mass Transfer Model for Basic Blue Adsorption onto Pillared Bentonite Clay Using Langmuir Equilibrium and Taking into Account Liquid and Surface Diffusion
Hadiatni Rita Priyantini, Wahyudi Budi Sediawan, Rochmadi and Imam Prasetyo PKM – 1
- 2 Evaluasi Teknis Separator Uap dan Pengaturan Temperatur Pada Mesin Pengereng kayu di Industri Pengolahan Kayu PT. Triowira Kalimantan.
Mamat PKM – 9
- 3 Penggantian *Purge gas Recovery unit* (PGRU) berbasis Teknologi Cryogenic menjadi berbasis Teknologi Membran di Pabrik Amoniak Pusri IV
Muhammad Natsir Hakiki, Ferlyn Fachlevie, Yusman Arullah PKM – 15
- 4 Evaluasi Unjuk Kerja Pengereng Hasil Pertanian Bawang Merah dengan Sistem Pemanfaatan Energi Terpadu Matahari dan Angin
Samsul Kamal PKM – 20

TEKNIK INDUSTRI

- 1 Model Prediksi Kesuksesan Produk Dengan Pertimbangan Pengaruh Kemudahan Inovasi
Ana Yunita Masura, Subagyo, dan Fitri Trapsilawati TI – 1
- 2 Korelasi Variabel Competitive Priorities dengan Keunggulan Bersaing Industri Di Provinsi Gorontalo
Iwan Inrawan Wiratmadja, Trifandi Lasalewo TI – 7
- 3 Karakterisasi Jaringan Sosial Difusi Teknologi Gadget
Khairunnisa dan Bertha Maya Sopha TI – 13
- 4 Pengukuran *Brand* dan Inovasi Untuk Prediksi Nilai *Intangible* Pada Penentuan Harga Produk
Reza Bayu Kurniawan dan Subagyo TI – 19
- 5 Peningkatan Efisiensi Pabrik Amoniak dan Urea di PT Pupuk Indonesia (Persero)
Sigit Abdurrakhman TI – 25
- 6 Pembuatan Perangkat Lunak Guna Otomatisasi Penggunaan *Control Chart*
Wilhelmus Abisatya Pararta, dan Hari Agung Yuniarto TI – 31
- 7 Pengembangan Framework dan Standard Operating Procedure (SOP) Perencanaan Turnaround Maintenance Berbasis Project Management
Zoelverdi Yustian Putra dan Hari Agung Yuniarto TI – 36

TEKNIK REAKSI DAN TEKNIK PEMBAKARAN

- | | | |
|---|--|------------|
| 1 | Pengaruh Suhu dan Kecepatan Pengaduk pada Reaksi Ozonolysis dan Transesterifikasi Minyak Goreng Bekas
<i>Lieke Riadi, Lie Hwa, Edy Purwanto dan Alloysius Yuli Widianto</i> | TR/TP – 1 |
| 2 | Analisis Termal Tungku Untuk Proses Produksi Carbon Black di PT Mocaf Indonesia Trenggalek
<i>Mamat, Bambang Prihandoko</i> | TR/TP – 7 |
| 3 | Studi Paduan U-Mo Monolitik Sebagai Pengembangan Bahan Bakar Dispersi Densitas uranium Tinggi Untuk Reaktor Riset
<i>Supardjo</i> | TR/TP – 13 |
| 4 | Studi Kinetika Kalsinasi Zircon Oxychloride (ZOC)
<i>Tundjung Indrati Y</i> | TR/TP – 21 |
| 5 | Kajian Kinetika Pelarutan Slurry Zirconiumhydroxide (ZOH)
<i>Tundjung Indrati Y</i> | TR/TP – 32 |

PERPINDAHAN KALOR DAN MASSA

Mass Transfer Model for Basic Blue Adsorption onto Pillared Bentonite Clay Using Langmuir Equilibrium and Taking into Account Liquid and Surface Diffusion

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Abstract

Bentonite clay from Pacitan, East Java, was made using intercalation and pillarization method, using cationic surfactant HDTMA-Cl as intercalating agent and NaOH – AlCl₃ mixture with OH/Al ratio was 0,8 as pillaring agent at 80°C. Furthermore, calcination was done at 500°C for 4 hours. Adsorption process was done at laboratory scale batch system with initial Basic Blue 41 dye concentration varied between 100 ppm, 200 ppm, 300 ppm, 400 ppm, and 500 ppm for adsorbent mass ratio was 0,1 g/L and adsorbent size of 100 mesh. Adsorbent mass ratio was varied 0,1 g/L; 0,2 g/L; 0,25 g/L; 0,35 g/L; and 0,4 g/L for initial dye concentration of 600 ppm. Adsorption equilibrium was analyzed using Langmuir equilibrium model, and kinetic model was developed with considering convection mass transfer at adsorbent surface and intra-particle dye diffusion. Mathematic model and experimental data was used to calculate dye convective mass transfer and effective diffusion coefficient using computer program. The result showed that Basic Blue 41 – pillarized bentonite clay equilibrium obeyed Langmuir equilibrium model. Developed kinetic model was relatively fitted to experimental data. Maximum dye removal was 97,947 %. Generally, Basic Blue 41 adsorption kinetic was controlled by film resistance at adsorbent surface and intra-particle diffusion resistance.

Keywords: Adsorption; Basic Blue; Bentonite Clay; Modeling; Pillarization

1. Introduction

Bentonite clay, which was composed of alumina-silicate, is a low price commodity, widely available, and considered to be an effective sorbent material having a high added value. Based on the mineral content, the clay can be divided into: smectite (montmorillonite), kaolinite, halloysite, chlorite and illite [Olphen et al., 1977]. Bentonite is a type of clay which has the ability to adsorb water and its cation exchange power much greater than the ordinary clay. A component of the dominant clay mineral in bentonite is montmorillonite. Montmorillonite has the chemical formula $[NaxAl(2-x)Mgx(Si_4O_{10})(OH)_2] \cdot (H_2O)_m$.

Montmorillonite clay minerals are a group that has a good ability to adsorb both the metal and the organic molecules. It also has an ability to inflate and deflate (swelling) and an exchangeable cation [Pinnavaia, 1983]. Bentonite is a clay composed of alumina-silicate which consists of 3 layers, where a layer containing octahedral aluminum flanked between the other two layers containing tetrahedral silicate. This three layers form a sheet structure [Astrology, 2000].

Due to its large porosity, beside its potential as an adsorbent, bentonite clay is very suitable to be used as catalysts and ion exchange material. However, although bentonite clay is very useful in many areas of application, it has limitations in maintaining its shape, due to its nature of easy to swell. Clays can swell due to hydration, deflate and even the structure of the clay can be damaged by dehydration and heating. To prevent this deformation, the interlayer region should be supported by a stable pillar. The other purpose of such modification is to create a large pore volume. In addition, it was also addressed to increase its ability of maintaining the porosity of clay despite hydration and dehydration occur. This process is called pillarization [Robert et al., 1999].

Two main steps of the pillarization process were as follows, 1). Inserting a pillaring agent to the layer between the clay sheets, 2). Calcining the material to make the pillaring agent incorporated and tied into the solid sheets strongly. The so-called pillaring agent is any substance that can form the pillars on sheets of clay, so the clay becomes porous. While, the insertion of these substances into the space between the sheets (interlayer) without changing the structure of the sheet is called intercalation stage. Bentonite that has undergone a process of pillarization is called pillared bentonite clay [Robert et al., 1999].

To form a stable pillared bentonite with permanent porosity, the process of heating (calcination) needs a great consideration. The best temperature range for calcination is between 300-500 °C [Barrer et. al, 1978]. Pinnavia described that the calcination process at the temperature range makes the pillaring agent strongly bind to the bentonite clay sheets. This is indicated by the spectral data A1 MAS-NMS on smectite clays pillared with Al (Barrer et al, 1978). After experiencing pillarization, the pillared bentonite clay has expands vertically compared to prior pillarization, and can not return to the original form despite the dehydration happen [Robert et al., 1999].

Besides being able to expand vertically due to dehydration, other characteristics of pillared bentonite is having basal spacing at least 0.315 to 0.353 nm. The pillaring agent molecules are laterally in the area of intermellar which has porous characteristic. However, the basic criteria of pillared bentonite is having a good thermal and chemical stability and having a good distribution of the pillars in the intermellar region [Robert et al., 1999].

The aim of this research work is to develop a mass transfer model which is suitable for basic blue adsorption onto pillared bentonite clay by taking into account the intra particle concentration gradient and applying linier equilibrium equation. The suitability of the model was examined by using the data of laboratory experiments and analyzed quantitatively.

2. Theory and Experimental

2.1 Modeling

Since the pillared bentonite clay particles used in this research work is sphere-like shape, it was considered appropriate to assume the shape of pillared bentonite to be spherical shape, then the mass transfer model was developed using volume element mass balance for sphere:

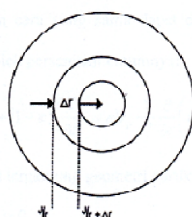


Figure 1. Volume element mass balance

$$\left(-D_e \cdot 4\pi \cdot r^2 \frac{\partial C}{\partial r} \Big|_r - D_{es} \cdot 4\pi \cdot r^2 \frac{\partial C_\mu}{\partial r} \Big|_r \right) - \left(-D_e \cdot 4\pi \cdot r^2 \frac{\partial C}{\partial r} \Big|_{r+\Delta r} - D_{es} \cdot 4\pi \cdot r^2 \frac{\partial C_\mu}{\partial r} \Big|_{r+\Delta r} \right) = 4\pi \cdot r^2 \Delta r \frac{\partial}{\partial t} (C_\mu \rho_{ads} + C\varepsilon)$$

$$D_e \left(\frac{\partial^2 C}{\partial r^2} + \frac{2}{r} \frac{\partial C}{\partial r} \right) + D_{es} \left(\frac{\partial^2 C_\mu}{\partial r^2} + \frac{2}{r} \frac{\partial C_\mu}{\partial r} \right) = \frac{\partial}{\partial t} (C_\mu \rho_{ads} + C\varepsilon) \quad (1)$$

If $q = C_\mu \rho_{ads} + C\varepsilon$ then

$$D_e \left(\frac{\partial^2 C}{\partial r^2} + \frac{2}{r} \frac{\partial C}{\partial r} \right) + D_{es} \left(\frac{\partial^2 C_\mu}{\partial r^2} + \frac{2}{r} \frac{\partial C_\mu}{\partial r} \right) = \frac{\partial q}{\partial t} \quad (2)$$

With

$$q = C_\mu \rho_{ads} + C\varepsilon$$

$$= \frac{\alpha C}{C + \beta} \rho_{ads} + C\varepsilon$$

$$Cq + \beta q = \alpha C + C^2 \varepsilon + \beta C \varepsilon \quad (3)$$

$$C^2 \varepsilon + (\alpha \rho_{ads} + \beta \varepsilon - q)C - \beta q = 0$$

Solution of the equation (3) is

$$C = \frac{-(\alpha\rho_{ads} + \beta\varepsilon - q) + \sqrt{(\alpha\rho_{ads} + \beta\varepsilon - q)^2 + 4\beta q\varepsilon}}{2\varepsilon} \quad (4)$$

Equation (2) is the general form of differential equation for adsorption of solute from liquid phase to solid sphere which was taken into account the intra-particle concentration gradient. Differential equation (2) was solved using finite difference approximation which expressed as:

$$\left[\left(\frac{i-1}{iM} \right) C_{i-1,j} - \frac{2}{M} C_{i,j} + \left(\frac{i+1}{iM} \right) C_{i+1,j} \right] + \left[\left(\frac{i-1}{iM'} \right) C_{\mu,i-1,j} - \frac{2}{M'} C_{\mu,i,j} + \left(\frac{i+1}{iM'} \right) C_{\mu,i+1,j} \right] + q_{i,j} = q_{i,j+1} \quad (5)$$

$$M = \frac{(\Delta r)^2}{D_e \Delta t} \quad \text{and} \quad M' = \frac{(\Delta r)^2}{D_{es} \Delta t} \quad (6)$$

For a certain position in spherical shape, it is necessary to modify the general finite difference equation as follow:

At the center of the sphere ($i = 0$)

$$\frac{D_e}{\alpha} (C_{1,j} - C_{0,j}) + \frac{D_{es}}{\alpha} (C_{\mu 1,j} - C_{\mu 0,j}) + q_{0,j} = q_{0,j+1} \quad (7)$$

At solid surface ($i = N$)

$$q_{N,j+1} = \left[\frac{\beta}{\alpha'} \left(D_e \frac{C_{N,j} - C_{N-1,j}}{(\Delta r)^2} + D_{es} \frac{C_{\mu N,j} - C_{\mu N-1,j}}{(\Delta r)^2} \right) + \frac{1}{\alpha} \frac{k_c 4\pi R^2 (C_{AL} - C_{\mu N,j})}{\Delta r} \right] \Delta t + q_{N,j} \quad (8)$$

Solute concentration in the liquid phase (C_f) was calculated using the equation:

$$C_{f,j+1} = C_{f,0} - \frac{4\pi N R}{V} \int_0^2 q dr \quad (9)$$

There are several methods available to solve the finite difference approximation such as explicit (forward), implicit (backward) and Crank-Nicolson [Wahyudi Budi Sediawan and Agus Prasetya, 1997]. In this research work, the finite difference approximation equation was solved using explicit method. The suitability of the model was examined by evaluating the sum of squares of errors (SSE) which was expressed as:

$$SSE = \sum (C_{\text{model}} - C_{\text{experimental}})^2 \quad (10)$$

2.2 Experiment

Bentonite clay, which was originated from Pacitan, East Java – Indonesia, was treated following intercalation and pillarization method. The measurement of some physical properties such as density, total porosity, macro and micro pore size and surface area BET (Brunauer, Emmet, Teller) was conducted afterward. Cationic surfactant (HDTMA-Cl) was used in intercalation while sodium hydroxide and $AlCl_3$ was used during pillarization process. The C.I. Basic Blue 41 under the trade name T/A Blue RGN-T 200% was used as adsorbate.

Intercalation is performed by preparing bentonite suspensions in which the ratio of bentonite/water = 1 gram/50 ml was applied and was diluted using distillate water to 1000 ml of total mixing volume. Surfactant was then added to the bentonite suspension and was stirred for 5 hours at a temperature of 80 °C. The weight ratio of surfactant/bentonite used was 1: 25. After being stirred for 5 minutes the solution was cooled briefly and was separated from the filtrate by a vacuum pump and then was dried in a laboratory scale oven at 100°C for 1 hour.

Pillaring agent was made by mixing NaOH and $AlCl_3$, in which the ratio of OH/Al was 0.8, at a temperature of 80 °C and was stirred until homogeneous mixing was reached. Intercalated bentonite suspensions was stirred and heated until the temperature reaches 80 °C. After that pillaring agent, ratio of Al/bentonite = 10 mmol/gram, was added gradually to a suspension of intercalated bentonite and stirred for 5 hours at 80 °C. After 5 hours, bentonite was separated from the filtrate using a vacuum pump and dried in an oven at 100 °C for 1 hour. Finally the bentonite was calcined at 500 °C in a laboratory scale furnace for 4 hours with a gradual increase to the calcination temperature every 15 minutes to avoid a collapse in the structure of pillared bentonite. To ensure uniformity, cool pillared bentonite powder then passed to a 100 mesh sieve prior the analysis of surface area BET and other

physical and chemical characterization. Pore radius, pore surface area and pore volume was analyzed using NOVA Data Analysis Package Ver. 2.00, Quantachrome Corporation.

Adsorption test of Al pillared bentonite powder was performed using an artificial waste which was prepared by dissolve a basic blue dye in 100 ml distilled water. Experiments were conducted by varying the initial dye concentration and bentonite weight / solution volume ratio. During the batch experiment, the dye concentration changes in liquid phase was detected by taking samples and measure the concentration using an UV spectrophotometer 600 nm.

3. Results and Discussion

The scanning electron microscope (SEM) image of original and pillared bentonite was shown on Figure 2 and Figure 3, respectively. The effect of the pillarization treatment was shown by the different surface appearance of the two images. The pillared bentonite image showed clearly the formation of basal spacing and area of intermellar where the pillaring agent molecules are laterally distributed.

The result of the composition analysis of the original and pillared bentonite was shown on Table 1. It could be seen from the table that the mass fraction of aluminum oxide increased to some significant extent, i.e. from 18.23 %w to 54.16 %w. The increasing aluminum oxide content is due to the infiltration of pillaring agent $AlCl_3$. Then, it indicated that the pillarization process has strongly incorporated and tied the pillaring agent into the bentonite solid sheets. While the mass fraction of some components including silicon dioxide, calcium oxide and ferro oxide decreased as consequent of increasing the aluminum oxide content.

Larger pore volume was detected on pillared bentonite which reflects increasing the surface area after pillarization. Accumulated pore volume and pore surface area were presented on Figure 4 and Figure 5, respectively. The figures showed clearly that the pillarization increased pore volume and pore surface, significantly. There has been a tremendous shift of pore size distribution dominates, from 0-20 Angstroms for the original bentonite to 20-500 Angstrom for pillared bentonite.

Figure 6 and Figure 7 displays basic blue concentration in the liquid phase by comparing the experimental data to the calculated value from the model for variations in the initial concentration. While Figure 8 and Figure 9 displays the same thing for variations in the ratio of mass of bentonite to volume of solution.

The parameters associated with the calculation of dye concentration, which include the distribution constant, external mass transfer coefficient and diffusivity coefficient, generated during solving the finite difference approximation were presented in Table 2 and Table 3.

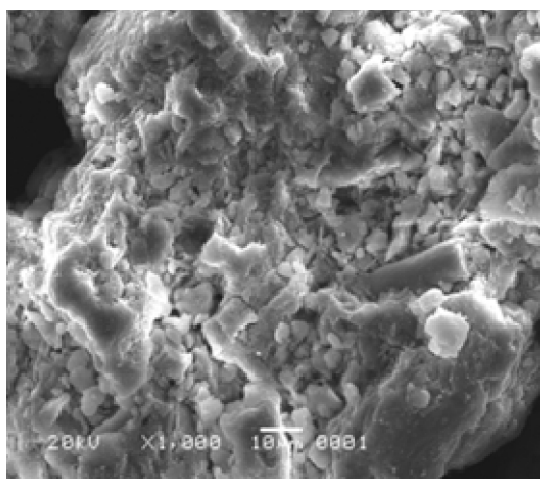


Figure 2: Scanning electron microscope image of original bentonite at magnification 1000x

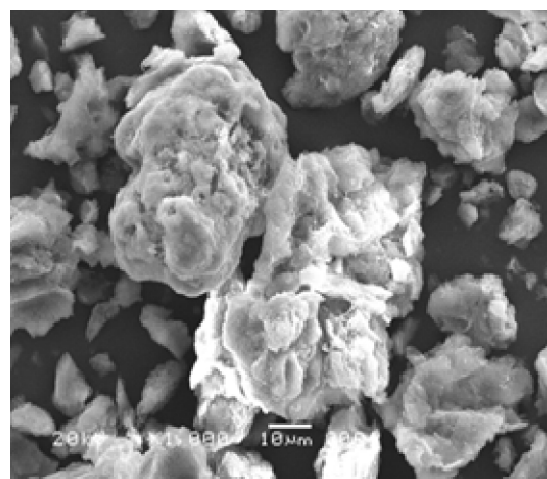


Figure 3: Scanning electron microscope image of pillared bentonite at magnification 1000 x

Table 1: Composition of Original and Pillared Bentonite

Component	Original bentonite , %w	Pillarred bentonite , %w
Na ₂ O	0,05	0.54
MgO	3.70	2.32
Al ₂ O ₃	18.23	54.16
SiO ₂	68.69	40.91
CaO	3.90	0.06
TiO ₂	0.59	0.16
FeO	4.83	1.85

Analysis methode : SNI 0449-2010

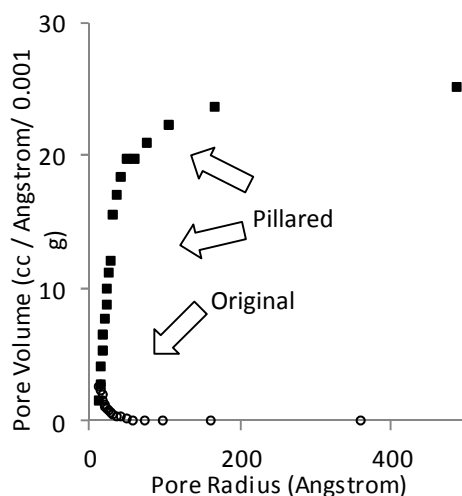


Figure 4: Accumulated pore volume versus pore radius of original and pillared bentonite clay.

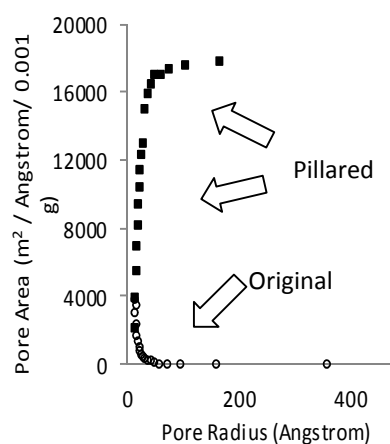


Figure 5: Accumulated pore surface area versus pore radius of original and pillared bentonite clay.

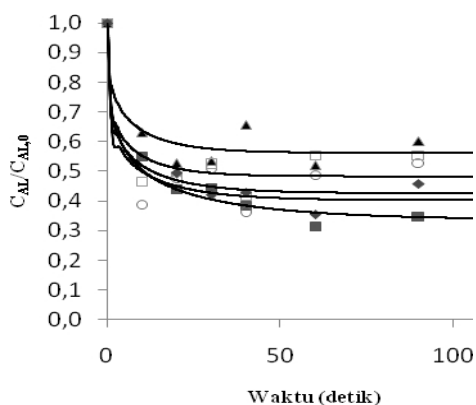


Figure 6: Basic blue concentration ratio in liquid phase versus time. The experimental data symbol for related initial concentration: ■ 100 ppm, ○ 200 ppm, ▲ 300 ppm, □ 400 ppm, ◆ 500 ppm. The curved line expressed the calculated result from the model. The mass ratio of bentonite/solution: 0.10 g/100 ml.

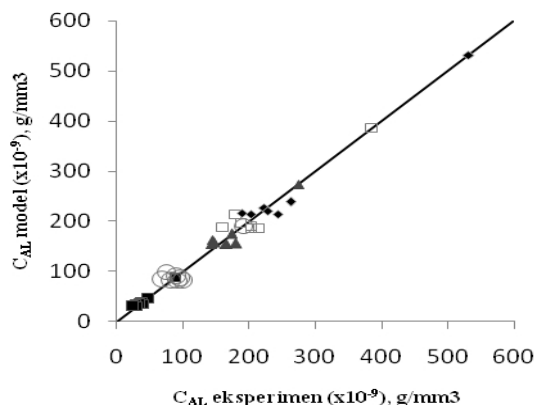


Figure 7: Basic blue concentration in liquid phase, model versus experimental data. The experimental data symbol for related initial concentration: ■ 100 ppm, ○ 200 ppm, ▲ 300 ppm, □ 400 ppm, ◆ 500 ppm. The mass ratio of bentonite/solution: 0.10 g/100 ml.

The distribution constants obtained from the experiments were in the range of $0.9 \cdot 10^{-5}$ - $1.9 \cdot 10^{-4}$ g/ml. Distribution constants for dye adsorption by bentonite was apparently not affected by the dye concentration in solution, while an increase in the ratio of bentonite/solution slightly lowering the distribution constant value as was shown on Table 2 and Table 3.

The external mass transfer coefficient (k_c) varies in the range of 3.0 – 5.5 cm/s. It wasn't found a clear correlation between the external mass transfer coefficient and the initial dye concentration as well as the bentonite/solution ratio.

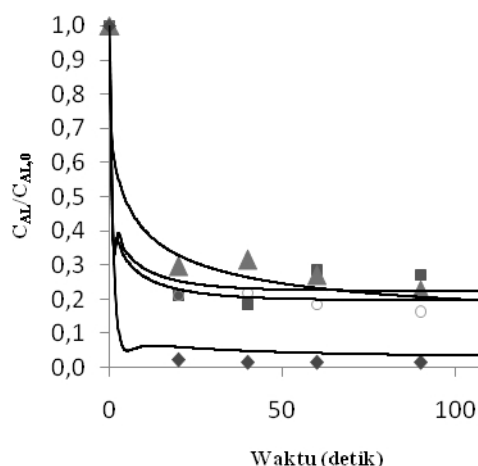


Figure 8: Basic blue concentration ratio in liquid phase versus time. The experimental data symbol for related bentonite/solution ratio: \square 0.20 g/100 ml, \blacklozenge 0.25 g/100 ml, \blacktriangle 0.35 g/100 ml, \circ 0.40 g/100 ml. The curved line expressed the calculated result from the model. The initial concentration of basic blue was 600 ppm.

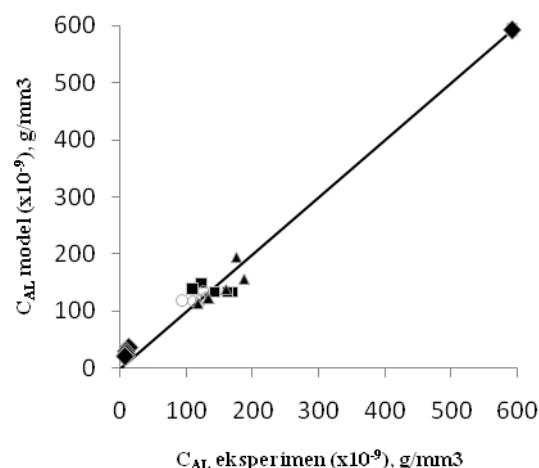


Figure 9: Basic blue concentration in liquid phase, model versus experimental data. The experimental data symbol for related bentonite/solution ratio: \blacksquare 0.20 g/100 ml, \circ 0.25 g/100 ml, \blacktriangle 0.35 g/100 ml, \blacklozenge 0.40 g/100 ml. The curved line expressed the calculated result from the model. The initial concentration of basic blue was 600 ppm.

Table 2: Distribution constant, external mass transfer coefficient, diffusivity coefficient and SSE at various initial dye concentration for bentonite/dye solution ratio 0.1g/100 ml

C_{i0} ppm	α L/mg	β mg/g	k_c cm/s	D_e cm^2/s	D_{es} cm^2/s	SSE
100	2.5	56.35	5.5	$6.6 \cdot 10^{-4}$	$1.00 \cdot 10^{-68}$	$6.7 \cdot 10^{-17}$
200	2.5	82.70	3.5	$1.0 \cdot 10^{-3}$	$1.00 \cdot 10^{-68}$	$1.5 \cdot 10^{-15}$
300	2.5	143.0	4.0	$8.0 \cdot 10^{-4}$	$1.00 \cdot 10^{-68}$	$1.4 \cdot 10^{-15}$
400	2.5	104.0	3.4	$1.1 \cdot 10^{-3}$	$1.00 \cdot 10^{-68}$	$3.8 \cdot 10^{-15}$
500	2.5	75.00	3.0	$1.0 \cdot 10^{-3}$	$1.00 \cdot 10^{-68}$	$2.3 \cdot 10^{-15}$

Table 3: Distribution constant, external mass transfer coefficient, diffusivity coefficient and SSE at various bentonite/dye solution ratio for initial dye concentration 600 ppm

B/D g/100 mL	α L/mg	β mg/g	k_c cm/s	D_e cm ² /s	D_{es} cm ² /s	SSE
0.2	3.2	82.7	3.8	1.2 10 ⁻³	1.00 10 ⁻⁶⁸	6.0 10 ⁻¹⁵
0.25	3.0	82.7	4.2	1.0 10 ⁻³	1.00 10 ⁻⁶⁸	4.7 10 ⁻¹⁵
0.35	4.2	82.7	5.0	1.7 10 ⁻⁴	1.00 10 ⁻⁶⁸	2.9 10 ⁻¹⁵
0.4	99	18.8	5.4	1.7 10 ⁻⁴	1.00 10 ⁻⁶⁸	2.2 10 ⁻¹⁵

Varying the initial concentration of dye at constant bentonite/solution ratio and varying the bentonite/solution ratio at constant initial dye concentration mean varying the dye concentration during the batch adsorption process.

While value of the external mass transfer coefficient calculated from the same experimental data source vary, as shown in Table 2 and Table 3. It mean that a change of dye concentration might be result in a change of an eddy diffusivity and/or the thickness of liquid phase interface film, since an external mass transfer coefficient depend on both the eddy diffusivity and the thickness of liquid phase interface film.

The sum of squares of errors which was used to examine the suitability of the developed model was presented in Table 2 for initial dye concentration variation and in Table 3 for bentonite/solution ratio variation. The value were in the range of 6.7 10⁻¹⁷ - 6.0 10⁻¹⁵. This low value of SSE indicates that the developed mass transfer model is suitable for basic blue adsorption onto pillared bentonite clay.

4. Conclusion

The effect of the bentonite pillarization was shown by the different surface appearance which was found on the scanning electron microscope images. The formation of basal spacing and area of intermellar was identified in the pillared bentonite.

After pillarization, the mass fraction of aluminum oxide increased to some significant extent, i.e. from 18.23 %w to 54.16 %w. The increasing aluminum oxide content is due to the infiltration of pillaring agent AlCl₃. It indicated that the pillarization process has strongly incorporated and tied the pillaring agent into the bentonite solid sheets.

Larger pore volume was indicated by pillared bentonite which reflects increasing the surface area after pillarization. There has been a tremendous shift of pore size distribution dominates, from 0-20 Angstroms for the original bentonite to 20-500 Angstrom for pillared bentonite.

This research work has proven that the developed mass transfer model are suitable for basic blue adsorption onto pillared bentonite clay.

Acknowledgements

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Nomenclature

- B/D* bentonite/dye solution ratio (g / 100 ml)
- C_f* solut concentration in liquid phase (mol/ml)
- C_{fo}* initial solut concentration in liquid phase (mol/ml)

C^*	solut concentration in liquid phase interface (mol/ml)
$C_{i,j}$	solut concentration at position i at time j
C_μ	solut concentration in solid (g/g solid)
D_e	pore diffusivity coefficient (cm ² /s)
D_{es}	surface diffusivity coefficient (cm ² /s)
k_c	external mass transfer coefficient (cm/s)
m_{in}	rate of mass inflow (mol/s)
m_{out}	rate of mass outflow (mol/s)
m_{acc}	rate of mass accumulated (mol/s)
N	total number of particle
R	sphere radius (cm)
r	distance from the sphere center in radial direction (cm)
t	time (s)
<i>Greek letters</i>	
ε	particle porosity
ρ_{ads}	adsorbent density (g/ml)
α	konstanta Langmuir (L/mg)
β	konstanta Langmuir (mg/g)

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