

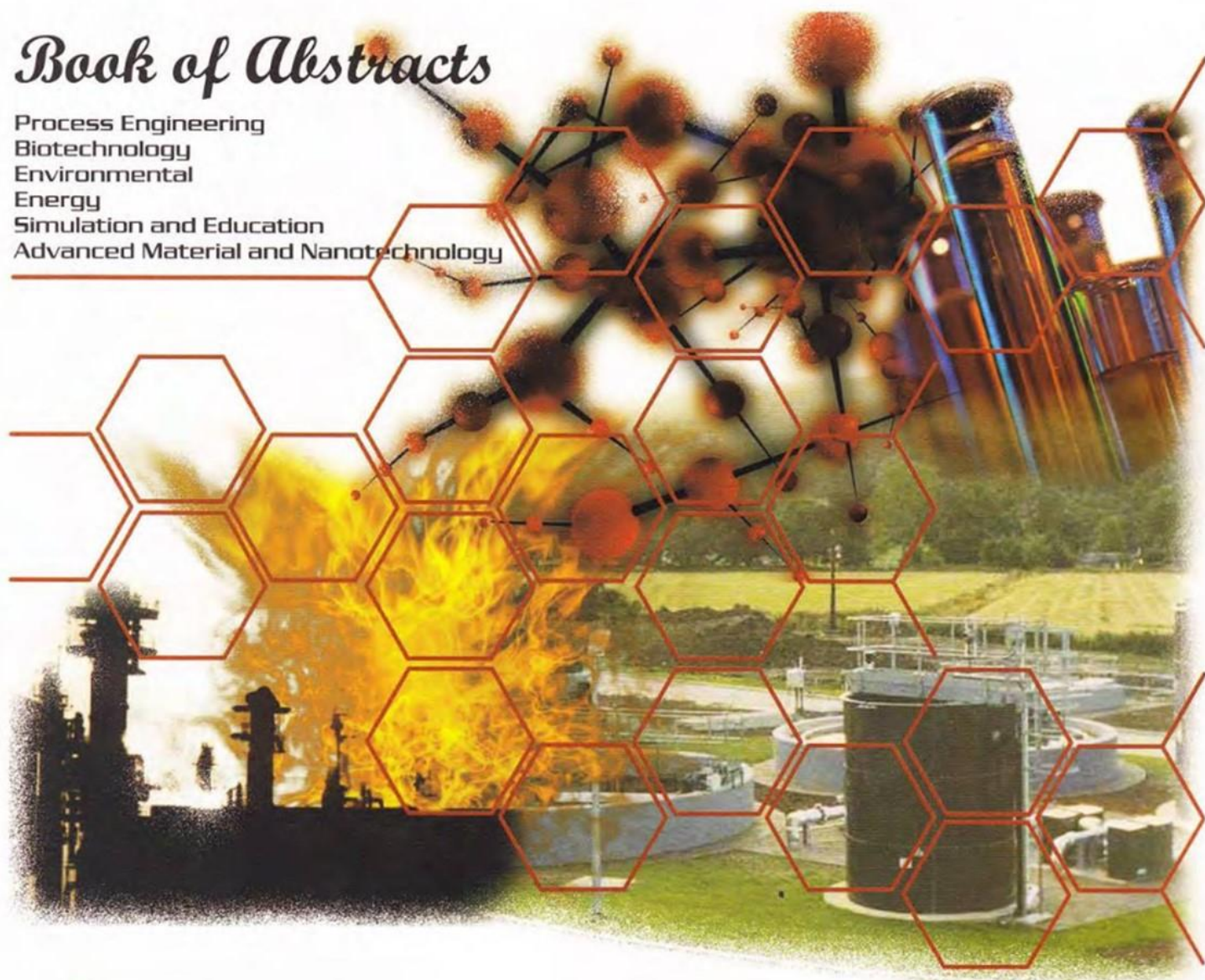
December 4-5, 2007  
Melia Purosani Hotel  
Yogyakarta - Indonesia

# RSCFE

## Regional Symposium on Chemical Engineering

### Book of Abstracts

Process Engineering  
Biotechnology  
Environmental  
Energy  
Simulation and Education  
Advanced Material and Nanotechnology



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## ABOUT RSCE 2007

The Regional Symposium on Chemical Engineering (RSCE) has become an important annual forum for academicians, researchers, and industrial practitioners in South East Asia and Asia Pacific region to exchange knowledge and information in the growing spectra of chemical engineering science and technology. This event has gained its reputation as a pivotal role in improving chemical engineering research and linkages with other relevant disciplines, especially in ASEAN region.

The previous symposia were held in the Philippines (1993, 1998, 2003), Thailand (1995, 1999, 2004), Indonesia (1996, 2001), Malaysia (1997, 2002), Vietnam (2005), and Singapore (2000, 2006). To continue the excellent tradition in promoting the exchange of ideas among people from a variety of institutions working in areas related to chemical engineering, The 14<sup>th</sup> RSCE 2007 is scheduled to be hosted by Gadjah Mada University, Indonesia on December 4-5, 2007.

More than 200 papers from academics/industrial practitioners have been received by the symposium secretariat to be presented in oral/poster presentations. The two day symposium covers the vast arrays of interests: Process Engineering Group, Environmental Group, Biotechnology Group, Energy Group, Simulation and Education Group and Advanced Materials and Nanotechnology Group.

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Yogyakarta, December, 4-5<sup>th</sup> 2007

Chemical Engineering Department, Gadjah Mada University



## Schedule For RSCE 2007 Yogyakarta, Indonesia

### Tuesday December, 4<sup>th</sup> 2007

Time	Schedule	Place
08.00 – 09.00	Registration	Front desk
09.00- 09.30	Opening	Amarta Ballroom
09.30 – 10.00	Plenary I	Amarta Ballroom
10.00 – 10.30	Plenary II	Amarta Ballroom
10.30 – 11.00	Coffee break	Coridor & around
11.00 – 11.30	Plenary III	Amarta Ballroom
11.30 – 12.00	Plenary IV	Amarta Ballroom
12.00 – 13.00	Lunch	Melia Resto
13.00 – 15.00	Presentation I	( see Note)
15.00 – 15.30	Coffee break	Coridor & Around
15.30 – 17.00	Presentation II	(see Note)
19.00 - 22.00	Gala Dinner <sup>*)</sup>	Amarta Ballroom

<sup>\*)</sup> Invitation only

### Wednesday, December, 5<sup>th</sup> 2007

Time	Schedule	Place
08.30 – 09.00	Plenary V	Amarta Ballroom
09.00- 09.30	Plenary VI	Amarta Ballroom
09.30 – 10.00	Coffee break	Coridor & Around
10.00 – 12.00	Presentation III	(see note)
12.00 – 13.00	Lunch	Melia Resto
13.00 – 15.00	Presentation IV	( see Note)
15.00 – 15.30	Coffee break	Coridor & Around
15.30 – 17.00	Presentation V	(see Note)

## SCHEDULE OF ORAL PRESENTATION

Tuesday,  
Dec 4, 2007

Time									
	Amarta Ballroom		Yudhistira	Bima	Arjuna	Nakula	Sadewa	SMR 1	SMR 2
08.00-09.00	Registration								
09.00-09.30	Opening Ceremony								
09.30-10.00	Plenary 1								
10.00-10.30	Plenary 2								
10.30-11.00	Coffee Break								
11.00-11.30	Plenary 3								
11.30-12.00	Plenary 4								
12.00-13.00	Lunch								
13.00-13.20	PE 1	PE 25	EV 16	BI 1	EG 1	SE 1	AM 1	PE 49	AM 31
13.20-13.40	PE 2	PE 26	EV 17	BI 2	EG 2	SE 2	AM 2	PE 50	AM 32
13.40-14.00	PE 3	PE 27	PE 58	BI 3	EG 3	SE 3	AM 3	PE 51	AM 33
14.00-14.20	PE 4	PE 28	PE 59	BI 4	EG 4	SE 4	AM 4	PE 52	AM 34
14.20-14.40	PE 5	PE 29	PE 60	BI 5	EG 5	SE 20	AM 5	PE 53	BI 21
14.40-15.10	Coffee Break								
15.10-15.30	PE 45	PE 30	EV 6	BI 6	EG 6	SE 10	AM 6	PE 54	PE 7
15.30-15.50	PE 46	PE 31	EV 7	BI 7	EG 7	SE 11	AM 7	PE 55	PE 6
15.50-16.10	PE 47	PE 32	EV 8	BI 8	EG 8	SE 12	AM 8	PE 56	PE 8
16.10-16.30	PE 48	PE 34	EV 9	BI 9	EG 9	SE 13	AM 16	PE 57	PE 9
16.30-16.50									

Wednesday,  
Dec 5, 2007

Time	Amarta Ballroom	Yudhistira	Bima	Arjuna	Nakula	Sadewa	SMR 1	SMR 2
08.30-09.00	Plenary 5							
09.00-09.30	Plenary 6							
09.30-10.00	Coffee Break							
10.00-10.20	PE 10	PE 33	EV 10	BI 10	EG 10	SE 6	AM 10	AM 25
10.20-10.40	PE 11	PE 35	EV 11	BI 11	EG 11	SE 7	AM 11	AM 26
10.40-11.00	PE 12	PE 36	EV 12	BI 12	EG 12	SE 8	AM 12	AM 27
11.00-11.20	PE 13	PE 37	EV 13	BI 13	EG 13	SE 9	AM 13	AM 28
11.20-11.40	PE 14	PE 38	EV 14	BI 14	EG 14	SE 14	AM 14	AM 29
11.40-12.00	PE 15	PE 39	EV 15	BI 15	EG 15	SE 15	AM 15	AM 30
12.00-13.00	Lunch							
13.00-13.20	PE 16	PE 40	EV 1	BI 16	EG 16	SE 16	AM 9	SE 25
13.20-13.40	PE 17	PE 41	EV 2	BI 17	EG 17	SE 17	AM 17	SE 26
13.40-14.00	PE 18	PE 42	EV 3	BI 18	EG 18	SE 18	AM 18	SE 27
14.00-14.20	PE 19	PE 43	EV 4	BI 19	EG 19	SE 19	AM 19	SE 28
14.20-14.40	PE 20	PE 44	EV 5	BI 20	EG 20	SE 5	AM 20	EG 25
14.40-15.10	Coffee Break							
15.10-15.30		PE 21	SE 29		SE 21	AM 21	EG 21	
15.30-15.50		PE 22	SE 30		SE 22	AM 22	EG 22	
15.50-16.10		PE 23	SE 31		SE 23	AM 23	EG 23	
16.10-16.30		PE 24	SE 32		SE 24	AM 24	EG 24	
16.30-17.00	Closing Ceremony							



## LIST OF ORAL PRESENTATION RSCE 2007

### PROCESS ENGINEERING GROUP

PE	Authors	Title
1	Abdurahman H. Nour, R. Mohd Yunus, A. Hasyam	Water-in-Crude Oil Emulsions: Its Stabilization and Demulsification
2	Aditya Putranto, Judy Reti Witono, Arina Purwanti, and Dian Nataka	Osmotic dehydration of <i>Mangifera indica</i> with Zugarramurdi and Lupin method
3	Agung Nugroho, Kiki Yustendi, and Tjandra Setiadi	The Effect Of Cod Concentration On Organic Acids Production From Cassava Ethanol Stillage
4	Aning Ayucitra, Chris Colby	Gelatinisation and Retrogradation Properties of Acetylated Corn Starches with Various Degrees of Substitution
5	Aswabi Mindaryani, Muhammad Febnan	Kinetics of Air Drying of Cassava starch
6	Bahrudin, Sumarno, Gede Wibawa, Nonot Soewarno	The effect of Maleated Polypropylene on the Morphology and Mechanical Properties of Dynamically Vulcanized Natural Rubber/Polypropylene Blends
7	Dewi Tristantini Øyvind Borg Børje Gevert, Anders Holmen	Effect Of Water Addition On Direct Use Of H <sub>2</sub> -Poor Bio-Syngas Model In Fischer-Tropsch Synthesis Over Co/Al <sub>2</sub> O <sub>3</sub> Catalyst
8	Dyah Setia Novianti, Yolanda P Brondial, Servittario Oiano Jr Junjro Kawasaki	Vapor-Liquid-Liquid Equilibria Of Acetone-Toluene-Water With And Without Salt Effect
9	Eden Marikit, Chris Salim and Hirofumi Hinode	Effect of Ionic Strength on the Adsorption and Photocatalytic Oxidation of Humic Acid at neutral Ph
10	Eka Prasetya Merdika, Joseph Aurensia, Raymond G.R. Tan, Masaaki Suzuki	Control System Design and Modeling on CNT Production using Modified Microwave Oven
11	Enjarlis, Setijo Bismo, Slamet Roekmijati	Kinetics Degradation of Carbofuran By Ozonation in presence of Activated carbon
12	Hayder A. Abdul Bari	Drag Reduction Improvement in Two Phase Flow System Using Traces Of Sles Surfactant
13	Heri Hermansyah, Masako Kubob, Naomi Shibasaki-Kitakawab, Toshikuni Yonemotob	Rigorous Kinetic Model for Triglyceride Hydrolysis Using Porcine Pancreatic Lipase

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|----|---|--|
| 13 | Tanyakorn Muangnapoh, Tawatchai Charinpanitkul and Noriaki Sano                         | Effect Of Ph On Stability Of Gold Nanoparticles Synthesized By Aqueous Reaction  |
| 14 | C.H.M. Citraningrum, Gunawan, N. Indraswati, Suyadi Ismadji                             | Adsorption Capacity Improvement Of Phenol Solution Onto Thermal Modified Granular Activated Carbon                                     |
| 15 | Weny Irawaty  | Non-Aqueous Solvothermal Route To Prepare Mono-Dispersed Titanium Dioxide Nanoparticles Suspension                                     |
| 16 | Watadta and Prasert Pavasant  | Characterization of Polyelectrolyte Multilayer-Deposited Electrospun Cellulose Acetate Fibrous Membrane                                |
| 17 | Yuswan Muharam, Widodo W. Purwanto and Anisa Afianty                                    | The Effect Of Textural Promoters On The Quantity And Quality Of Nanocarbons Through Methane Decomposition Using Ni/Cu-Based Catalysts  |
| 18 | Yuswan Muharam, Widodo W. Purwanto and Anisa Afianty                                    | Production Of Carbon Nanotubes And Hydrogen From Methane Decomposition In The Reactor With A Structured Catalyst                       |
| 19 | Samsudin Affandi, Heru Setyawan, Dwiatri, Dewi Handriyani and Sugeng Winardi            | A Simple Low-Energy Method To Produce Silica Gel With High Surface Area From Bagasse Ash   |
| 20 | Yupin Phuphuak, Keisuke Fukaya, and Nurak Grisdanurak                                   | Effect Of Ni Doping In Ce-Mg-O Nanosize Catalysts For CO Oxidation   |
| 21 | Ratna Balgis, Dyan Wardani, Ahmad Fadli, Heru Setyawan, and Sugeng Winardi              | Synthesis Of Mesoporous Silica By Gelatin-Templated Sol-Gel Method From Water Glass  |
| 22 | Heru Setyawan, Minta Yuwana and Sugeng Winardi  | Synthesis Of Spherical And Donut-Shaped Silica Particles Derived From Water Glass-Based Colloidal Nanoparticles By Spray Drying Method |
| 23 | Arief Budhyantoro, Restu Kartiko Widi   | Sinthesys Of Organoclay From Natural Bentonit  |
| 24 | Suryadi Ismadji   | Influence Of Pore Size Distribution And Surface Chemistry On The Adsorption Of Organic Compounds Onto Activated Carbon                 |
| 25 | Tarinee Nampitch  | Natural Rubber/Layered Silicate Nanocomposites : A Review From Preparation To Processing   |
| 26 | Pranuda Jivaganont, Pimpa Limthongkul, Sumittra Charojrochkul, Rapeepong Suwanwarangkul | Microstructural Investigation In Anode-Supported Sofc Via A Micro Model  |
| 27 | Budiyono And Luqman Buchori   | The Influence Of Casting Machine Speed In Membrane Preparation   |
| 28 | Liza Nazifah  | Tempering Of Coated And Uncoated Glass Experiment And Modelling  |



## ADSORPTION CAPACITY IMPROVEMENT OF PHENOL SOLUTION ONTO THERMAL MODIFIED GRANULAR ACTIVATED CARBON

AM 14

Marlistya Citraningrum, Gunawan, Nani Indraswati, Suryadi Ismadji

The adsorption capacity of activated carbon is not only influenced by the geometrical heterogeneity (porosity), but also by the chemical (functional groups fixed in carbon surface) or often called the surface chemistry. This surface chemistry can be modified using acid or thermal treatment in order to improve the acidity or basicity of the carbon. The main objective in this study is to modify a commercial granular activated carbon in order to produce a specific carbon for phenol adsorption. Phenol is one of common contaminants found in wastewater. A NORIT granular activated carbon was chosen as parent carbon, and then the modification was done by means of thermal treatment under nitrogen flow at various temperatures. Different techniques (determination of acidity, basicity, and the pH at the point of zero charge,  $N_2$  adsorption at 77 K, XRD, and SEM) were used to characterize the carbons. The treatment caused the basic groups of the carbon increased in a significant number. Pore structure and pore size distribution do not undergo any significant changes after thermal treatments have been done. Then the adsorption capacity was studied by adsorption of phenol at different pH of solution. The different uptakes obtained are discussed in relation to the surface chemical properties of the carbons and the influence of solution pH. It was found that the higher the treatment temperature, the more basic the surface of activated carbon, and the more phenols can be absorbed. This conclusion is explained based on dispersive interactions. Moreover, it also shows that the adsorption process is better when pH of solution is above  $pH_{PZC}$ , related with the dissociation of hydrophilic surface groups, reducing the uptakes of water complexes. The adsorption isotherm data can be represented by Dubinin – Raduskevich (DR) adsorption isotherm.

*Keywords: activated carbon, phenol, surface chemistry, thermal treatment*

## NON-AQUEOUS SOLVOTHERMAL ROUTE TO PREPARE MONODISPERSED TITANIUM DIOXIDE NANOPARTICLES SUSPENSION

AM 15

Wenny Irawaty

Titanium dioxide ( $TiO_2$ ) has a potential application in advanced technologies such as solar cell, photo-induced selfcleaning, photoelectrochromic windows, gene therapy, catalysis, drug delivery, degradation of organic contaminants, photocatalysis, etc. Due to these wide applications, the study of  $TiO_2$  has attracted increasing attention and a number of  $TiO_2$  nanostructures, including nanoparticles, nanorods, nanotubes, nanowires, hollow spheres have been reported to date. In this research, for coating application, we aim to prepare the transparent suspension of titanium dioxide containing nearly mono-dispersed nanoparticles. Non-aqueous solvothermal method is chosen to obtain high crystalline of  $TiO_2$  at low temperature. The solvothermal reactions are carried out in a sealed autoclave that provides suitable conditions for hydrolysis, polycondensation reactions and formation of nanoparticles. The suspension was prepared by stirring ammonium bicarbonate, triethylamine, ethyl acetate and linoleic acid in a flask. Titanium tetra-butoxide was added dropwise into the solution and stirred for 5 minutes. Different volume of linoleic acid was added. Then the solution was transferred and sealed in a stainless autoclave and heated at different temperatures for 24 hours without stirring. The collected products of  $TiO_2$  were characterized using X-ray diffractions (XRD), Zetasizer and TEM (Transmission Electron Microscopy). The results showed that the increase of temperature from 130 to 180°C and linoleic acid from 7 to 25 mL promote the colour of the suspensions are darker. Since ester  $C_{17}H_{31}COOC_4H_9$  was formed, the average particle size is increased with the volume of linoleic acid. The amount of catalyst promotes the nanoparticles reactions. A better suspension is obtained by adding triethylamine as catalyst. The suspension color is transparent and the particle size is between 21 and 27 nm. XRD patterns showed that this nanoparticles have a good structure, i.e. anatase. The stable and transparent suspension was obtained by non-aqueous route at 150°C. This suspension can be applied for coating purpose.

*Keywords: titanium dioxide, suspension, coating, non aqueous, solvothermal*

# NON-AQUEOUS SOLVOTHERMAL ROUTE TO PREPARE MONO-DISPERSED TITANIUM DIOXIDE NANOPARTICLES SUSPENSION

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## ABSTRACT

Titanium dioxide ( $\text{TiO}_2$ ) has a potential application in advanced technologies such as solar cell, photo-induced self-cleaning, photoelectrochromic windows, gene therapy, catalysis, drug delivery, degradation of organic contaminants, photocatalysis, etc. Due to these wide applications, the study of  $\text{TiO}_2$  has attracted increasing attention and a number of  $\text{TiO}_2$  nanostructures, including nanoparticles, nanorods, nanotubes, nanowires, hollow spheres have been reported to date. In this research, for coating application, we aim to prepare the transparent suspension of titanium dioxide containing nearly mono-dispersed nanoparticles. Non-aqueous solvothermal method is chosen to obtain high crystalline of  $\text{TiO}_2$  at low temperature. The solvothermal reactions are carried out in a sealed autoclave that provides suitable conditions for hydrolysis, polycondensation reactions and formation of nanoparticles.

The suspension was prepared by stirring ammonium bicarbonate, triethylamine, ethyl acetate and linoleic acid in a flask. Titanium tetra-t-butoxide was added dropwise into the solution and stirred for 5 minutes. Different volume of linoleic acid was added. Then the solution was transferred and sealed in a stainless autoclave and heated at different temperatures for 24 hours without stirring. The collected products of  $\text{TiO}_2$  were characterized using X-ray diffractions (XRD), Zetasizer and TEM (Transmission Electron Microscopy).

The results showed that the increase of temperature from 130 to 180°C and linoleic acid from 7 to 25 mL promote the colour of the suspensions are darker. Since ester  $\text{C}_{17}\text{H}_{31}\text{COOC}_4\text{H}_9$  was formed, the average particle size is increased with the volume of linoleic acid. The amount of catalyst promotes the nanoparticles reactions. A better suspension is obtained by adding triethylamine as catalyst. The suspension color is transparent and the particle size is between 21 and 27 nm. XRD patterns showed that this nanoparticles have a good structure, i.e. anatase. The stable and transparent suspension was obtained by non-aqueous route at 150°C. This suspension can be applied for coating purpose.

**Keywords:** titanium dioxide, suspension, coating, non aqueous, solvothermal

## I. INTRODUCTION

Titanium dioxide ( $\text{TiO}_2$ ) is a promising metal oxide that has a potential application in advanced technologies such as solar cell, photo-induced self-cleaning (hydrophilicity property), photoelectrochromic windows, gene therapy, catalysis, drug delivery, degradation of organic contaminants, and photocatalysis [1]–[5]. Due to these applications, the study of  $\text{TiO}_2$  has attracted increasing attention and a number of  $\text{TiO}_2$  nanostructures, including nanoparticles, nanorods, nanotubes, nanowires, hollow spheres have been reported to date [6]–[7].

Currently, developed synthetic routes for producing metal oxide nanoparticles are sol-gel process [8]–[9], microemulsion-assisted process [10] and non-aqueous solvothermal methods [6],[11]. The requirement of high crystallinity of metal oxide nanoparticles is a major problem in their synthesis. Sol-gel methods, included both hydrolytic and non hydrolytic, and microemulsions provide amorphous metal oxide. Calcination of gels is required to induce crystallization that leading to grain growth and loss of surface area. Hydrothermal synthesis solves the problems encountered during sol-gel process, but the product nanocrystals are rather agglomerate after a long (3 days) or short (from 5 minutes to a few hours) aging at moderate (140°C) or high temperatures (250–300°C) [7]. Commonly, some binders are added to form a stable colloidal solution. The binders are easily lumped with the  $\text{TiO}_2$  colloidal particle. After calcination step, the remained binders will form byproduct in VOC decomposition [12]. Non-aqueous solvothermal is the best method to get high quality of nanoparticles. In a sealed vessel, solvent is heated to temperature higher than its boiling point by increasing pressures in heating process [10]. The solvothermal



treatment can be used to control the particle's size, particle's morphology, and crystalline phase by varying the composition, temperature, solvents and aging time [11]. The solvothermal method is chosen to obtain highly crystalline  $\text{TiO}_2$  at low temperature. The solvothermal reactions are carried out in a sealed autoclave that provides suitable conditions for hydrolysis, polycondensation reactions and formation of nanoparticles [6].

Some kinds of nanoparticles are already available commercially in powders or liquid dispersions. The latter is obtained by combining nanoparticles with an aqueous or organic liquid to form a suspension or paste. It may be necessary to use chemical additives (surfactants, dispersants) to obtain a uniform and stable dispersion of particles. Further processing steps, nanostructured powders and dispersions can be used to fabricate coatings and paints. Reference [6] synthesized crystalline, nearly mono-dispersed and transparent solution of  $\text{TiO}_2$  in cyclohexane using solvothermal method.

In this research, for coating application, we aim to prepare the transparent suspensions of titanium dioxides containing nearly mono-dispersed nanoparticles by employing a non-aqueous solvothermal method. Based on the environment effect and healthy [13], ethyl acetate is chosen as solvent in this coating suspension preparation.

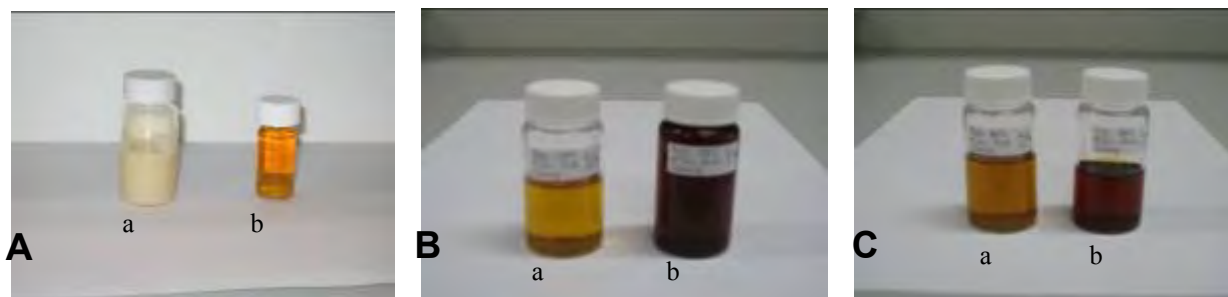
## II. METHODOLOGY

Reagents used for preparing  $\text{TiO}_2$  suspension were as follows: titanium tetra-*t*-butoxide (Aldrich 24,411-2), linoleic acid (Sigma L1626), ammonium bicarbonate (Sigma A6141), triethylamine (Merck M.8.08352) and ethyl acetate (Lab-Scan M.8.08352). All chemicals were used without any further purification.

The suspension was prepared by stirring ammonium bicarbonate, triethylamine, ethyl acetate and linoleic acid in a flask. Titanium tetra-*t*-butoxide was added dropwise into the mixed solution and stirred for 5 minutes. Then the solution was transferred and sealed in a stainless autoclave and heated at some different temperatures for 24 hours without stirring. The collected products of  $\text{TiO}_2$  were characterized using X-ray diffractions (XRD), Zetasizer and Transmission Electron Microscopy (TEM). The XRD pattern was recorded in the  $2\theta$  range from 20 to  $70^\circ$  with a Rigaku-MiniFlex at a scan speed of 2 degree per minute. Zetasizer (Malvern, nano ZS) has been used to get the particles size distribution and the micrograph of nanoparticles obtained using TEM (JEOL, JEM-1011).

## III. RESULTS AND DISCUSSION

The volume of linoleic acid (LA) as surfactants and temperatures were varied to study the effect of these variables on  $\text{TiO}_2$ . The obtained suspensions are shown in Fig. 1.

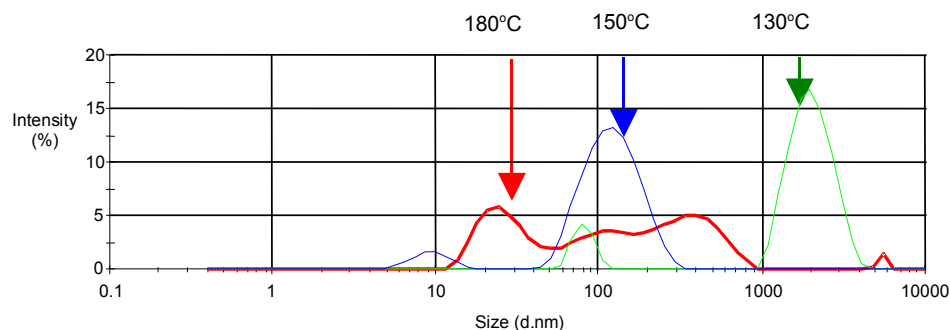


**Fig. 1**  $\text{TiO}_2$  suspensions at different volume of LA (mL): (a) 7; (b) 25 and temperatures ( $^\circ\text{C}$ ): (A) 130; (B) 150 and (C) 180

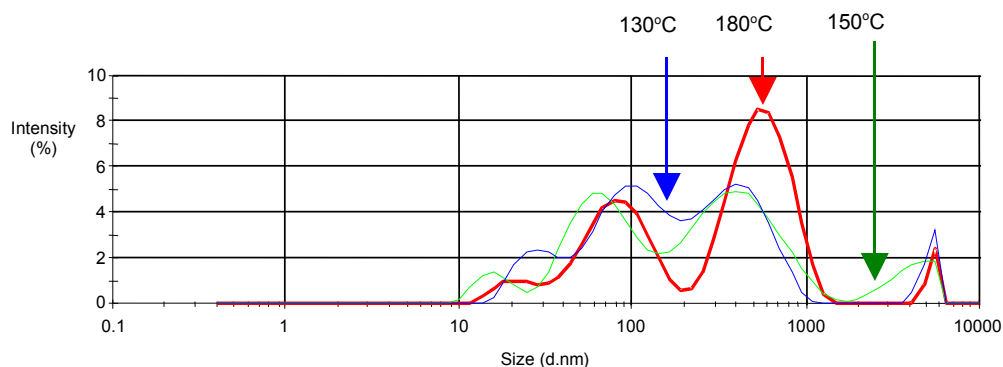
As can be seen from Fig. 1 that the increase of LA from 7 to 25 mL cause the suspensions were darker. LA,  $\text{C}_{17}\text{H}_{31}\text{COOH}$ , is one of polyunsaturated fatty acids. As a result, the heating process cause the fatty acid is unstable and the oxidation reaction promotes the suspension colors were darker. The oxidation process enhanced strongly with the increase of temperatures. The more LA added to the solution, the darker the suspension obtained.

Particle size distribution of  $\text{TiO}_2$  synthesized by solvothermal route at different volume of LA and temperatures are shown in Fig. 2 and 3. The average size of nanoparticles and suspension colors are shown in Table 1. Figure 2 and 3 show distributions of particles size obtained from Zetasizer at LA addition of 7 and 25 mL, respectively. The narrow particle size distribution was obtained at 7 mL of LA. As shown in Table 1, the average particle size at 7 mL LA was smaller than 25 mL and the particle size was increased with temperature, except data at  $130^\circ\text{C}$  – 7 mL LA. The morphology of nanoparticles did not affected by the volume of LA. At high temperature, ammonium bicarbonate will be decomposed to water, carbon dioxide and ammonia. Titanium precursor was hydrolyzed and promotes the  $\text{TiO}_2$  formation. LA will disperse  $\text{TiO}_2$  particles well.  $\text{TiO}_2$  precursor also reacts with LA and water to form  $\text{TiO}_2$  and  $\text{C}_4\text{H}_9\text{OH}$ . Since the volume of LA was increased,  $\text{C}_4\text{H}_9\text{OH}$  reacted with LA to form esters  $\text{C}_{17}\text{H}_{31}\text{COOC}_4\text{H}_9$  [6]. As a consequence, the average of particle size was increased with the volume of LA. The rate of chemical reaction is highly dependent upon temperature. Increasing the temperature from 130 to  $150^\circ\text{C}$  promote the  $\text{TiO}_2$  formation reactions. For temperature over  $150^\circ\text{C}$ , the formation of  $\text{TiO}_2$  and ester  $\text{C}_{17}\text{H}_{31}\text{COOC}_4\text{H}_9$  are faster so the average particle sizes were bigger as shown in Table 1. There was no good  $\text{TiO}_2$  suspensions produced from these synthesizes because large particles allowed the precipitation.

The increase of particle size can be seen clearly from the TiO<sub>2</sub> suspension colors as shown in Table 1. The changes of color from transparent to yellow colloidal showed that the particle size was increased. The yellow colloidal also indicated that the reactions were not completely at low LA and temperature. Extra time was needed to complete the reactions [11].



**Fig. 2** Particle size distributions of TiO<sub>2</sub> at addition of 7 mL LA with different temperature



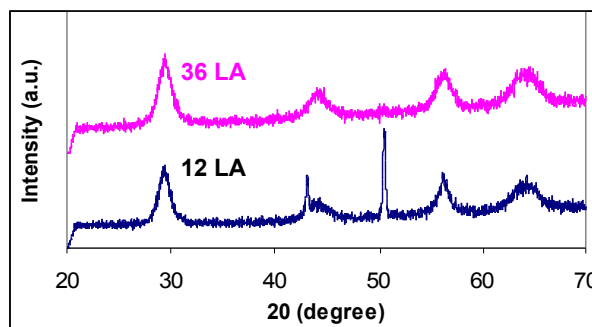
**Fig. 3** Particle size distributions of TiO<sub>2</sub> at addition of 25 mL LA with different temperature

**Table 1** TiO<sub>2</sub> particle size and suspension colors at different volume of LA and temperature

LA (mL)	Temperature (°C)	Particle size (nm)	Suspension
7	130	1,069	Yellow colloidal
	150	79	Brown*, transparent, precipitate
	180	110	Brown**, precipitate
25	130	135	Brown, transparent, precipitate
	150	113	Brown***, transparent, precipitate
	180	180	Brown***, precipitate

Note: the increase of \* shows the colour was darker

X-ray diffraction pattern of nanoparticles produced at 180°C and different volume of LA are shown in Fig. 4.

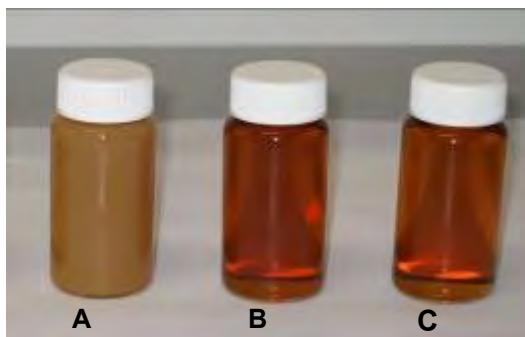


**Fig. 4** TiO<sub>2</sub> X-ray diffraction pattern at different volume of LA (mL)



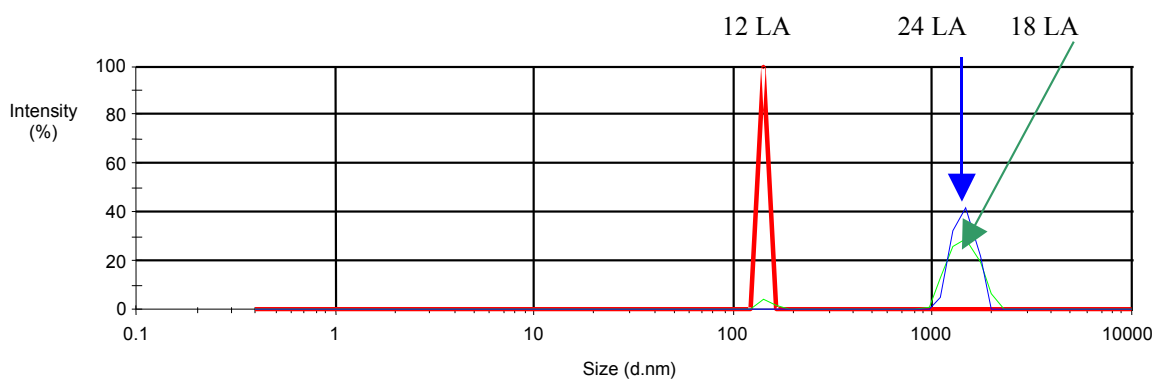
The reflection peaks as shown in Fig. 4 show that the nanoparticles had a sharp peak in the range  $2\theta$  between  $28^\circ$  and  $30^\circ$ . This obviously indicate the presence of crystalline phase of  $\text{TiO}_2$ . The crystalline phase was identified as the anatase phase.  $\text{TiO}_2$  nanoparticles synthesized with 36 mL of LA had a slightly sharp peak than the other. This indicate that the nanoparticles had a high crystallinity. The mechanical property of particles is affected by crystallinity degree. Besides as a surfactant, LA also improved the crystallinity of nanoparticles. This result is consistent with the previous study [6].

Based on the previous discussion, the best temperature for  $\text{TiO}_2$  nanoparticles formation was at  $150^\circ\text{C}$ . This temperature will be used for further research and the volume of LA still be varied to get a better suspension. The volume of LA was varied in the range between 12 and 24 mL.  $\text{TiO}_2$  suspensions produced at  $150^\circ\text{C}$  and different volume of LA are shown in Fig. 5.



**Fig. 5**  $\text{TiO}_2$  suspensions with different volume of LA: (A) 12; (B) 18 and (C) 24 mL

Fig. 5 shows that the increases of LA promote the nanoparticles reactions that affect the suspension colors. The effect of the volume of LA to particle size has discussed well. The particle size distributions of  $\text{TiO}_2$  are shown in Fig. 6. The average particle size and suspensions produced from these experiments are shown in Table 2.



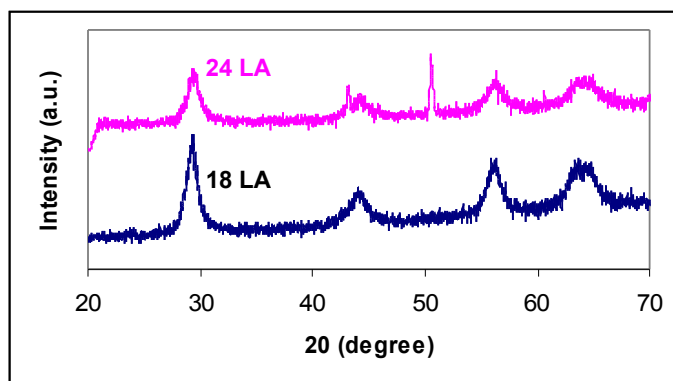
**Fig. 6** Particle size distribution prepared from different volume of LA (mL)

**Table 2**  $\text{TiO}_2$  particle size and colors obtained at  $150^\circ\text{C}$  and different volume of LA

LA (mL)	Particle size (nm)	Suspension
12	2,210	Brown colloidal
18	1,370	Brown*, transparent, precipitate
24	2,030	Brown*, transparent, precipitate

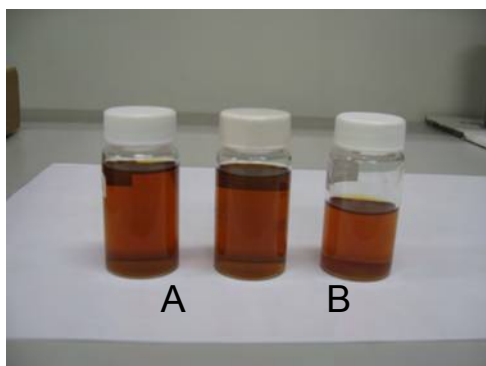
As can be seen from Table 2 that the average particles in suspensions were quite big that allows precipitation. The addition of 12 mL of LA might cause the reactions were not completely. As a consequence, the colloidal particles were produced. A better suspension was obtained from 18 and 24 mL of LA, despite there were some precipitate in suspensions. The smallest particle size was obtained by adding 18 mL of LA to the solution.

XRD pattern of  $\text{TiO}_2$  nanoparticles produced at  $150^\circ\text{C}$  in ethyl acetate solution at different volume of LA is shown in Fig. 7. A sharp peak was obtained by adding 18 mL of LA, this indicated that particles have a high crystallinity.



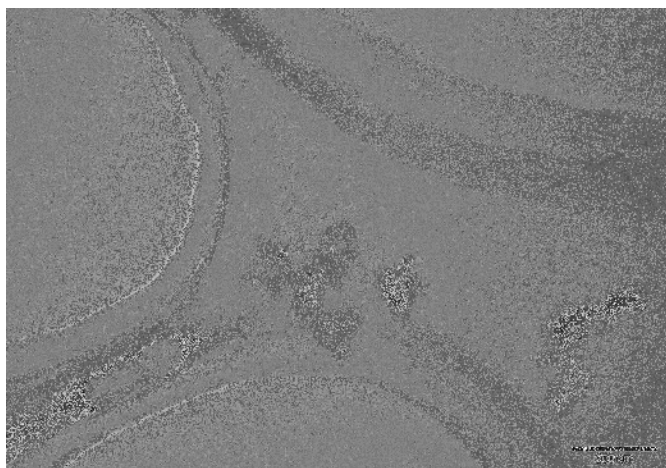
**Fig. 7** XRD pattern of TiO<sub>2</sub> nanoparticles at 150°C and different volume of LA (mL)

Based on the information in terms of average particle size, suspension color and XRD pattern, the optimum volume of LA in ethyl acetate solution was 18 mL. Compared with the previous results, the addition of more solvent in solution had not improved the suspension quality well. Another way that can be used to improve the process was by adding the volume of triethylamine (TEA) as catalyst. The increase of the catalyst amount might promote the nanoparticles reactions were better. In these experiments, the volume of TEA was doubled. The results are shown in Fig. 8.



**Fig. 8** TiO<sub>2</sub> suspensions with 10 mL of TEA and different volume of LA: (A) 18 and (B) 24 mL

The solutions were transparent but there was a little precipitates in the last one. It can be summarized that the best TiO<sub>2</sub> suspension was obtained by adding 18 mL of LA and 10 mL of TEA into ethyl acetate solution. The average particle size is between 21 and 27 nm. All the nanoparticles dispersed well. The TEM image of these nanoparticles is shown in Fig. 9.



**Fig. 9** TEM micrograph of TiO<sub>2</sub> nanoparticles with magnification x100,000



#### IV. CONCLUSIONS

In summary, the stable and transparent suspension was synthesized by non-aqueous solvothermal route at 150°C. TiO<sub>2</sub> suspension color was brown. The surfactant plays a key role in both color and nanoparticle size. TiO<sub>2</sub> nanoparticles obtained from this synthesis have anatase structure and the average particle sizes are between 21 and 27 nm.

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# CERTIFICATE

This is to certify that

**Wenny Irawaty**

has participated in the  
**14<sup>th</sup> Regional Symposium on Chemical Engineering**  
**December 4 - 5, 2007**  
**Yogyakarta - Indonesia**

As **Participant**  
(Presenter)

Committee Chairman



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