## สัญลักษณ์

สัญลักษณ์ที่ใช้ในภาคผนวก ก และ ข ตัวอักษรแสดงถึงชนิดของไซเลนที่ใช้ทำ ปฏิกิริยาซิลิเลชัน ตัวเลขแสดงถึงความเข้มข้นของสารละลายไซเลน และเวลาที่ใช้ในการทำ ปฏิกิริยาซิลิเลชัน ตามลำดับ ดังนี้

T = ปรับสภาพด้วย TMCS

D = ปรับสภาพด้วย DEDCS

ตัวเลขหลักที่ 1 = ความเข้มข้นของสารละลาย (ร้อยละ โดยน้ำหนัก)
ตัวเลขหลักที่ 2 = เวลาที่ใช้ในการทำปฏิกิริยา (ชั่วโมง)

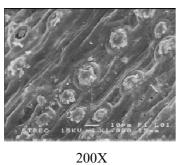
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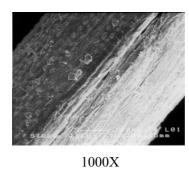
T13 = เส้นใยที่ปรับสภาพด้วย TMCS ความเข้มข้นร้อยละ 1 โดยน้ำหนัก ใช้เวลา ในการทำปฏิกิริยา 3 ชั่วโมง

#### ภาคผนวก ก

ภาพถ่าย SEM ของเส้นใยทะลายปาล์มเปล่าสดและที่ปรับสภาพด้วยวิธีซิลิเลชันที่ สภาวะต่างๆ กำลังขยาย 200 1000 5000 เท่า ตามลำดับ

## เส้นใยทะลายปาล์มเปล่าสด

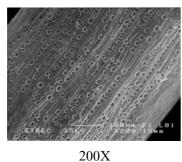


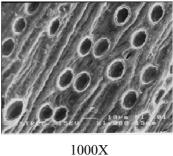




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## เส้นใยทะลายปาล์มเปล่าที่ปรับสภาพขั้นต้นด้วยวิธีเมอร์เซอร์ไรเซชัน (5%NaOH)

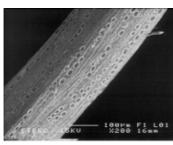


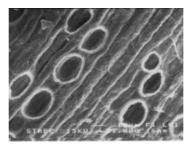


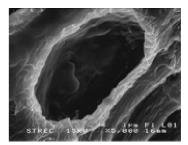


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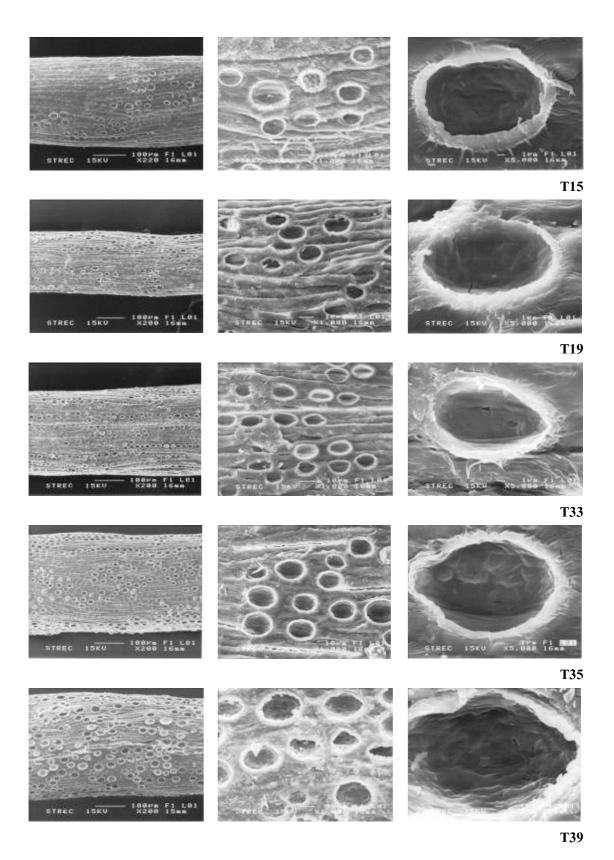
# เส้นใยทะลายปาลุ่มเปล่าที่ปรับสภาพด้วย TMCS



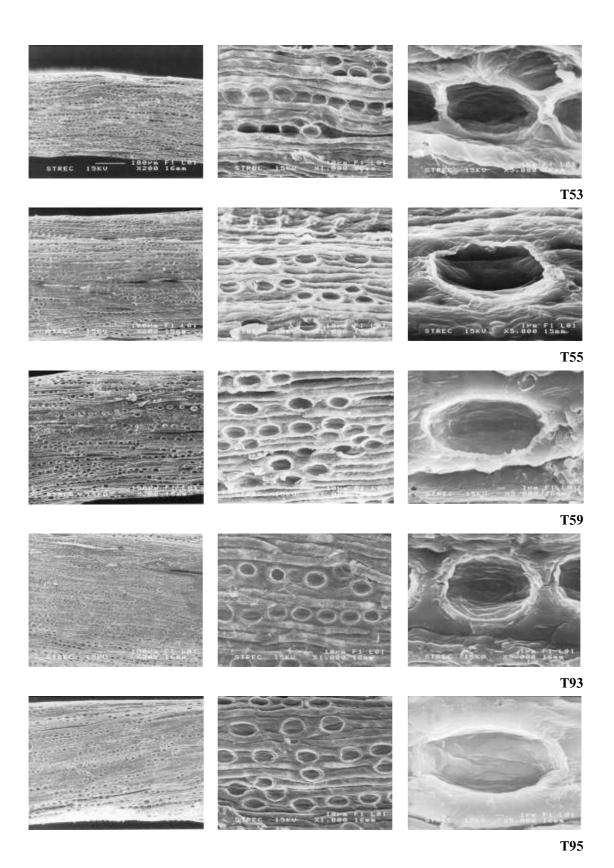


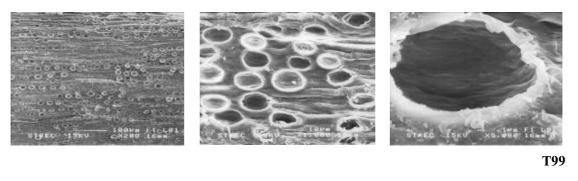


T13



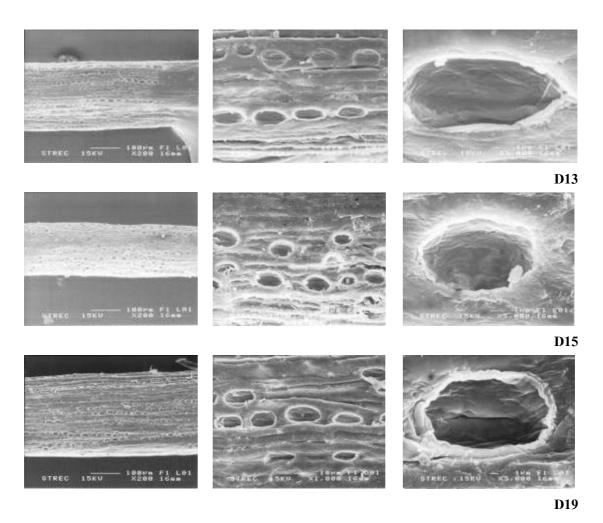
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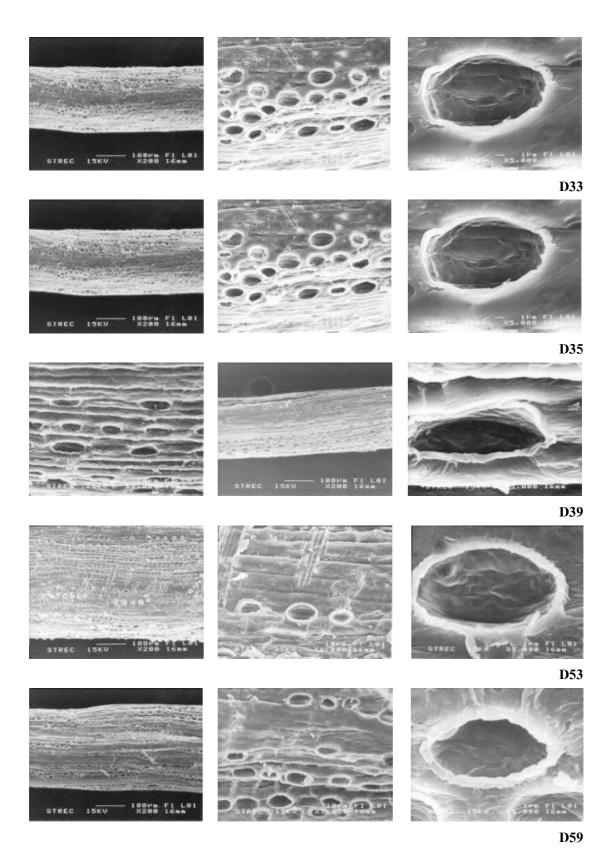


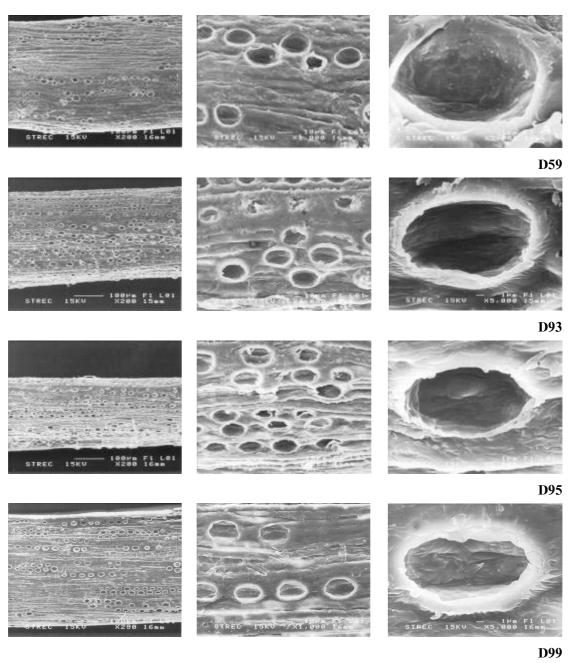
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# เส้นใยทะลายปาล์มเปล่าที่ปรับสภาพด้วย DEDCS



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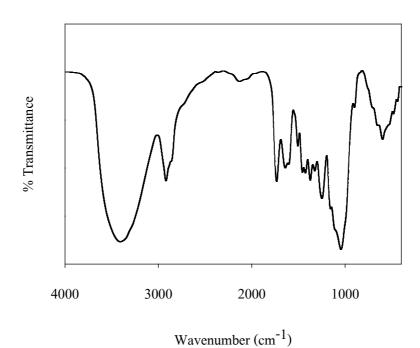




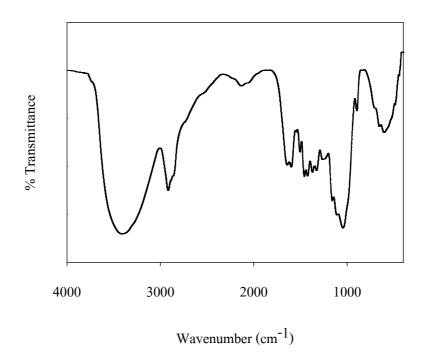
### ภาคผนวก ข

อินฟราเรคสเปกตรัมของเส้นใยทะลายปาล์มเปล่าสด และที่ปรับสภาพด้วยวิธี ซิลิเลชันที่สภาวะต่างๆ

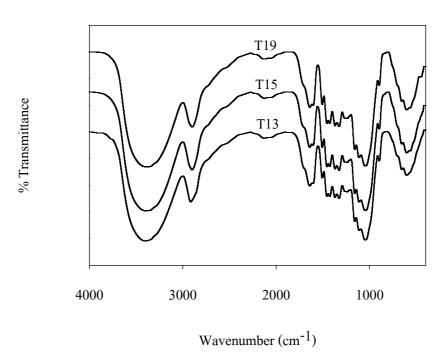
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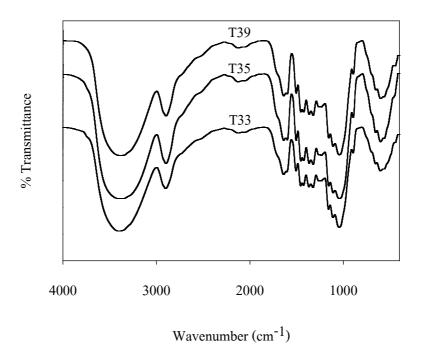


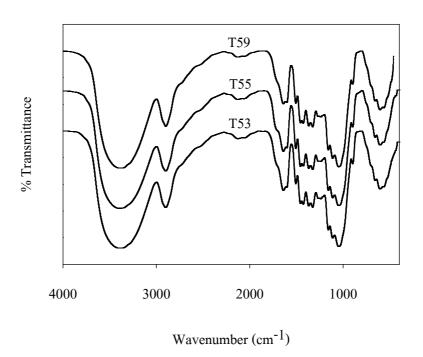
# เส้นใยทะลายปาล์มเปล่าที่ปรับสภาพขั้นต้นด้วยวิธีเมอร์เซอร์ไรเซชัน (5%NaOH)

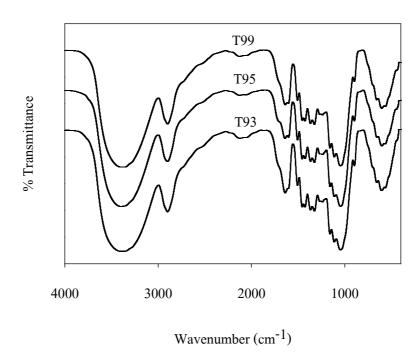


# เส้นใยทะลายปาลุ่มเปล่าที่ปรับสภาพด้วย TMCS

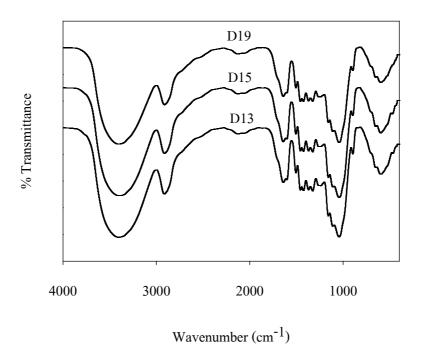


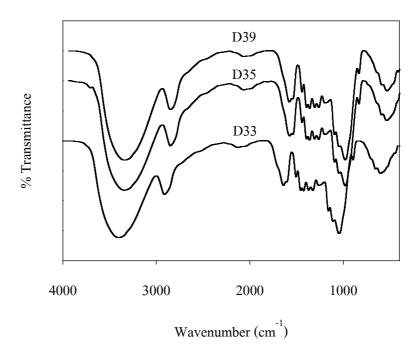


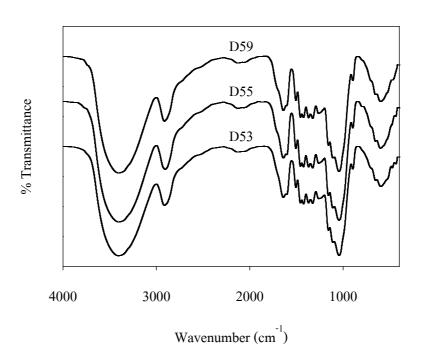


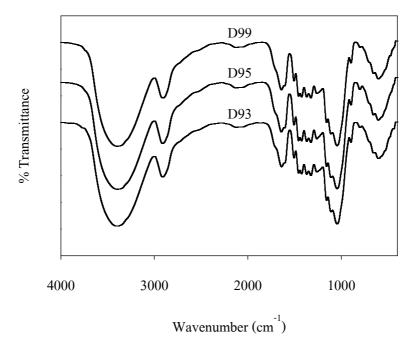


# เส้นใยทะลายปาล์มเปล่าที่ปรับสภาพด้วย DEDCS





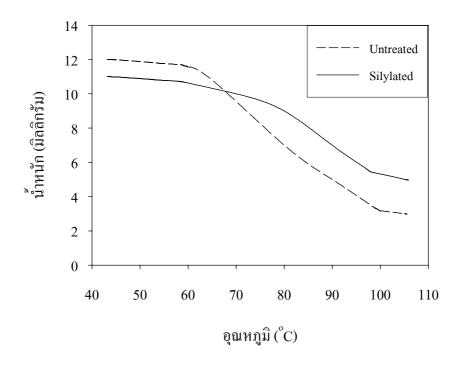




#### ภาคผนวก ค

## 1. การคำนวณปริมาณการดูดซับน้ำ

การวิเคราะห์โดยใช้เครื่องมือเทอร์โมกราวิเมทริกซ์ (Thermogravimetric analysis: TGA) เป็นการวัดค่าการเปลี่ยนแปลงน้ำหนักของตัวอย่างโดยการเพิ่มขึ้นของอุณหภูมิ ได้ทำการ ทดลองที่อุณหภูมิ 45 จนถึง 105°C น้ำหนักที่อ่านได้ตอนเริ่มต้น เป็นน้ำหนักของเส้นใยทะลาย ปาล์มเปล่าที่คูดซับน้ำไว้ เมื่ออุณหภูมิเพิ่มขึ้นน้ำหนักของเส้นใยจะค่อยๆ ลดลง เนื่องจากน้ำภายใน เส้นใยระเหยออกไป จนกระทั่งน้ำหนักเส้นใยคงที่ที่อุณหภูมิ 105°C เนื่องจากน้ำภายใน เส้นใย ระเหยออกหมด ซึ่งถือเป็นน้ำหนักของเส้นใยก่อนการคูดซับ แสดงได้ดังรูป



ปริมาณการคูดซับน้ำของเส้นใยทะลายปาล์มเปล่า คำนวณได้จากกราฟ ยกตัวอย่าง การหาปริมาณการคูดซับน้ำของเส้นใยทะลายปาล์มเปล่าสด (Untreated) จากกราฟอ่านค่าน้ำหนัก ได้ดังนี้

น้ำหนักเริ่มต้น ประมาณ	12	มิลลิกรัม
น้ำหนักสุดท้าย ประมาณ	3	มิลลิกรัม

น้ำหนักน้ำที่คูดซับได้ = 12 - 3 = 9 มิลลิกรัม ปริมาณการคูดซับน้ำของเส้นใยทะลายปาล์มเปล่าสด (กรัมน้ำต่อกรัมเส้นใย) =  $\frac{(1x9)}{3} = 3$  กรัม

## 2. การคำนวณปริมาณน้ำมัน

การวิเคราะห์หาปริมาณน้ำมันอาศัยหลักการดังนี้ คือ ปรับสภาพตัวอย่างที่เป็นของ เหลวให้เป็นกรด (พีเอชน้อยกว่า 2) เพื่อให้ไขมันและน้ำมันแตกตัวจากน้ำและทำให้แยกจากน้ำโดย การกรอง นำมาสกัดด้วยเครื่องมือสกัดซอกฮ์เลต โดยใช้เฮกเซนเป็นตัวทำละลาย จากนั้นจึงนำเฮก เซนที่มีไขมันและน้ำมันละลายอยู่ไประเหยจนแห้ง ชั่งน้ำหนักตะกอนที่เหลือซึ่งเป็นไขมันและน้ำ มันในตัวอย่าง

การเก็บตัวอย่างไม่ควรเก็บมาแล้วแบ่งวิเคราะห์ ตัวอย่างที่ยังไม่ได้วิเคราะห์ใน ทันที่จะทำการเก็บรักษาไว้ด้วยกรดกำมะถันเข้มข้นในอัตรา 2 มิลลิลิตรต่อตัวอย่างน้ำ 1 ลิตร แล้วเก็บไว้ที่อุณหภูมิ -4°C

การคำนวณหาปริมาณน้ำมันในสารผสมอิมัลชันระหว่างน้ำมันกับน้ำ หาได้จาก สมการดังนี้

ใขมันและน้ำมัน (กรัม/ลิตร) = 
$$\frac{\text{(B-A)}}{\text{V}} \text{x} 10^3$$

โดยที่ A = น้ำหนักขวดสกัดก่อนทำการสกัด (กรัม)

B = น้ำขวดหลังการสกัด (กรัม)

V = ปริมาตรน้ำตัวอย่าง (มิลลิลิตร)



MOST



Regional Symposium on Chemical Engineering 2005



**New Trends in Technology** towards Sustainable Development

# **Proceedings**

November 30th - December 2th, 2005

Hanoi University of Technology in collaboration with Ho Chi Minh City University of Technology





# Regional Symposium on Chemical Engineering 2005 RSCE 2005

#### "New Trends in Technology towards Sustainable Development"

RSCE 2005 Secretariat HANOI UNIVERSITY OF TECHNOLOGY Dai Co Viet Road 1, Hanoi, VIETNAM Tel/Fax: + 84 4 868 0070 Email: rsce@mail.hut.edu.vn

Website: http://www.hut.edu.vn/conference/rsce.htm

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Hanoi, 21st November, 2005

#### **INVITATION LETTER**

Dear Ms ORAWAN RATTANAWONG,

It is our great pleasure to invite you to attend the Regional Symposium on Chemical Engineering 2005, organized by Hanoi University of Technology in collaboration with Hochiminh City University of Technology. This symposium will be held on 30th November – 2nd December 2005 at Hanoi Horison Hotel, Hanoi, Vietnam.

We are very pleased to let you know that your presentation entitled "Hydrophobicity Enhancement of Natural Palm Fiber Modified by Trimethylcholrosilane" will be presented as being scheduled in the enclosed RSCE 2005 Program.

We are looking forward to seeing you in Hanoi.

Sincerely yours,

Prof Ha Duyen Tu

Chairman of the Regional Symposium on Chemical Engineering 2005 (RSCE 2005)

[OSP12]	Hydrophobicity Enhancement of Natural Palm Fiber Modified by Trimethylcholrosilane	266
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[OSP16]	Effect of Ethyl Acetate on the Emulsification Characteristics of Ethanol Blended with Diesel Fuel	283
[OSP17]	Study on the preparation of the hydrogenation catalysts and hydrogenation process of the used lube oil for getting the base lube oil with higher quality	288
[OSP18]	Hydrocracking of petroleum residue oil over aluminum borate supported nickel-molybdenum catalyst	293
[OSP19]	Synthesis of Structured Lipid 1,3-dicapryloyl-2-γ-linolenoyl-glycerol	298
[OSP20]	Kinetics and thermodynamics parameters of kapok seed oil extraction using ethanol in the presence of acetic acid	303

		Institute of Energy Research, Daejon, Korea
10:15 - 10:30	[OCA05]	Direct Hydroxylation of Benzene to Phenol with Hydrogen Peroxide Catalyzed by Fe/TiO <sub>2</sub>
		Garun Tanarungsun, Suttichal Assabumrungrat, Worapon Kiatkittipong, Piyasan Praserthdam, Hiroshi Yamada and Tomohiko Tagawa; Chulalongkorn Univ., Bangkok, Thailand; Silpakorn Univ., Nakorn Pathom Thailand; Nagoya University, Chikusa, Nagoya, Japan
10:30 - 10:45	[OCA15]	A Comparative Study of Liquid Phase Hydrogenation on Pd/SiO <sub>2</sub> in Organic Solvent and under Pressurized Carbon Dioxide
		Kunnika Phandinthong, Joongjai Panpranot, Wandee Luesaiwong, and Piyasan Praserthdam; Chulalongkom University, Bangkok, Thailand; Ministry of Science and Technology, Bangkok, Thailand
10:45 - 11:00	[OCA33]	Hydrogenation of Red Palm Oil Model Compounds Using Ru-TPPTS and Rh-TPPTS Water-Soluble Homogeneous Catalysts in Biphasic Systems
		T. J. L. Sitohang, IGBN Makertihartha, H.J. Heeres; Institut Teknologi Bandung, Indonesia
11:00 - 11:15	[OCA35]	Synthesized Al-MCM-22 Zeolite for Alkylation Benzene Process
		Tran Thi Nhu Mai, Nguyen Thi Ha, Nguyen Huu Bao, Vo Thi Lien, Pham Hung Son, Nguyen Ba Trung; Hanoi Univ. of Sciences, Vietnam Nat. Univ.; PetroVietnam; Danang Univiversity

## [Thursday, December 01, 2005]

## Section Organic Chemical Engineering

Parallel Session 03

13:30 - 13:45	[OCA09]	Physical Granulation of Mixed NPK Fetilizers
		I Dewa Gede Arsa Putrawan and D. Widyastri; Institut Teknologi Bandung, Indonesia
13:45 - 14:00	[OCA26]	Characterization of MgO- and TiO <sub>2</sub> -Supported Pt-Ru Catalyst and Activities for Ethylene Hydrogenation and n-Butane Hydrogenolysis
		S. Chotisuwan, J. Wittayakun, and B. C. Gates; Prince of Songkla University, Thailand
14:00 - 14:15	[OSP05]	Study on $C_{12}\text{-}C_{54}$ ester derived from Palm Kernel oil for drilling fluid formulation
		Ismail Mohd Saaid, Faidzal Haslah, Anwar Raja Daud Mohamad and Mohd Ismail Omar.; Universiti Teknologi PETRONAS, Malaysia
14:15 - 14:30	[OSP11]	Investigation on the flooding of the C-5 Column of the Dinhco Gas Processing Plant for the improvement of liquid productivity
		Phan Dinh Tuan, Ngo Xuan Hung; Hochiminh City University of Technology, Petrovietnam Manpower Training College, PETROVIETNAM
		PETROVIETNAM

14:30 - 14:45	[OSP16]	Effect of Ethyl Acetate on the Emulsification Characteristics of Ethanol Blended with Diesel Fuel
		Thanyatom Isarachewa, Apanee Luengnaruemitchai and Samai Jai-In; The Petroleum and Petrochemical College, Bankok, Thai land; Eng. Development Dept., Naval Eng. Command, Thailand
14:45 - 15:00	[OSP12]	Hydrophobicity Enhancement of Natural Palm Fiber Modified by Trimethylcholrosilane
		Rattanawong, O., Kaewsichan, L.1, Grisdanurak, N.; Prince of Songkla University, Thailand; Thammasat University, Thailand
15:00 - 15:30		Coffee break and Poster presentation
15:30 - 15:45	[OSP08]	Synthesis of Chromane Derivatives via Metal-mediated Intramolecular Addition to Carbonyl Groups
		Nguyen Van Cuong and Han K-Y; Ho Chi Minh University of Industry, Ho Chi Minh City, Vietnam; Chungbuk National University, Korea
15:45 - 16:00	[OSP15]	Preliminary Study of Fungal Resistance on Rubberwood using Wood Vinegar
		Thanate Ratanawilai, Somchai Chuchom, Sukritthira Ratanawilai, Sriwan Srisai; Prince of Songkla University, Thailand
16:00 - 16:15	[OSP09]	Synthesis of Nonionic Surfactant Alkyl Polyglucosides (APGs) Using Microwave Assisted Fischer's Two Stages Method
		Pham Thanh Quan, Nguyen Tran Khai, Nguyen Minh Nhat, Tran Thi Kim Ngoc; Ho Chi Minh city University of Technology, Vietnam
16:15 - 16:30	[OSP20]	Kinetics and Thermodynamic Parameters of the Kapok Seed Oil Extraction Using Ethanol in the Presence of Acetic Acid
		Yohanes Sudaryanto, Felycia Edy S., Wenny Irawaty, Anton Putro; The Catholic University of Widya Mandala Surabaya, Indonesia

#### Hydrophobicity Enhancement of Natural Palm Fiber Modified by Trimethylchlorosilane

Rattanawong, O.1, Kaewsichan, L.1, Grisdanurak, N.2.\*

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#### Abstract

Empty fresh fiber of palm was chosen for removing oil contaminated in waste effluent. It has been improved its adsorption by a silylation technique. After the treatment, hydroxyl groups of over natural fibers were removed and reduced. This leaded to enhance hydrophobicity of fibers was approximately 0.616 compared with untreated fiber that has hydrophobicity 0.195. The results from TGA disclosed that the strength of fiber was still remained as original one, while SEM showed more porosity occurred through modified fibers.

#### Keywords

Palm fiber; hydrophobicity; silylation.

#### Introduction

The effluent from fish-shrimp processing and palm oil industrial is mainly oily wastes. Mostly, oily contaminants exist in small dispersed droplets, which is difficult to recovery. When it is sent to WWT plant, this pollution reduces the performance of purification plants, leading to more severe impact. Adsorption process has been recommended as one of promising techniques to reduce such concentration before flowing through the treatment [1, 2]. Many researches have been revealed the performance of studied adsorbents and reported the development of adsorption procedure both through adsorbent and process. Natural fiber, such as oil palm fiber, is one of abundant agricultural wastes in the local southern area nearby the treated needed. However, OPEF possess hydrophilic properties lead to natural oil palm fibers likely adsorb water more than nonpolar compounds. To overcome the problem of oil contaminated reduction in waste effluent and also utilize agricultural waste, it is needed to investigate the possibilities of hydrophobicity enhancing of oil palm fibers.

Oil palm is one of important economic plants in the south of Thailand. The Oil palm plantation in the southern region has been increased every year. In 2003, it was found that more than 2 M-rai used for oil palm plantation [3]. For every ton of fresh fruit bunch (FFB), approximately 0.28 ton of empty fruit bunch (EFB) is remained as solid waste [4]. EFB has been utilized in several purposes including agricultural activities, and combustible fuel for boiler. More than 60% of EFB waste still remains unused [5]. Natural fiber is composed of lignocellulose material consisting of cellulose, hemicellulose, and lignin. The combination of cellulose and hemicelluloses are called holocellulose and usually account for 65-70% of the plant dry weight [6], which is the major component of plant fiber. These compositions are rich in hydroxyl groups, which are responsible for moisture sorption through hydrogen bonding. So, they possess hydrophilic properties that must be modified before utilizing for oil removal. Several techniques of hydrophobicity enhancement, such as acetylation, mercerization, and silylation, have been presented. Recently, few works on hydrophobicity enhancement by silylation has been disclosed.

Biomass sorbents including kapok fiber, cattail fiber, Salvinia sp., wood chip, rice husk, and bagasse were tesed for oil removal from gas station runoff. It was reported that more than 70% of oil concentration could be reduced by biomass sorbents [7]. Besides, oil adsorping filter from reed canary grass, flax, or hemp fiber showed the possibility for the oil adsorption [8].

EFB has been widely studied as an additive for a composite material [9, 10]. It was found that after modification with silylation would strengthen material by less water adsorption [9, 10]. Up to the present, there is no research carried out for EFB as an oil removal adsorbent. In this study, attempts to

enhance EFB for nonpolar compound sorption by silylation would be revealed. Chemical (by FTIR), thermal (by TGA) properties and structure images were used to verify our results. Eventually, was comparative adsorptions over water, oil, and mixtures of those were also presented.

#### Material and Methods

Fresh palm fiber was obtained from local palm mill (Univanich Palm Oil Public Co., Ltd., Krabi province located in the southern part of Thailand). It was dried at 60°C over night. Silylating agents including Trimethylcholrosilane (TMCS) and mercerizing agent as NaOH were used as treating agents.

#### Fiber Surface Modification

Two kinds of chemical modifications (mercerization and silylation) were used to study. Mercirization was used of the comparison work. Conditions on this method followed Sreekala et al [9, 10]. Fibers were dipped in 5% NaOH at room temperature for 48 h. The fibers were then washed in distilled water until washing solution was neutral. Fiber was then filtered and finally dried at 60°C.

Silylation: Fibers were dipped in 1% TMCS solution in toluene for about 3 hour at room temperature. After the reaction, silane treated fiber was washed with toluene, filtered and dried in an oven at 60°C overnight and kept in a desiccator.

Characterizations were carried out by FTIR, TPD, and SEM.

#### Hydrophobicity Testing and Applications

About 0.5 g of fibers were dipped in beaker containing with 40 ml of hexane and agitated with magnetic stirrer for about 10 minutes. The sorbent was next retrieved, allowed to drain for 3 minutes, and weight. The results are expressed in term of the proportion of gram hexane to gram water. This value is an estimation of the degree of hydrophobicity of the fibers.

#### Results and Discussion

#### 1. ThermalDecomposition of EPF

EPF mainly composes of cellulose, hemicellulose, and lignin. Before using as a adsorbent, it really needed to understand the amount of water containing. This base amount would use to compare with the further adsorption after the treating. The characterization was carried out in a thermal gravimetric technique studied from 40-550°C with controlled heating rate. The weight loss profiles were obtained. Additionally it was computed in term of differential mass proportional to temperature obtained for TPD spectrum. Figure 1 shows the data of untreated EPF after drying at 60°C.

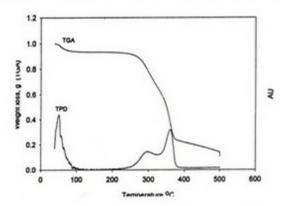


Figure 1. Composition of untreated EPF after drying at 60°C

Three steps of weight loss had been presented; obviously TPD spectrum was found three main peaks. Their maximum positions were located at 60, 295, and 360°C. The first peak, ranged from 40-100°C with centered at 55°C, is associated with physically adsorbed water. It seemed that water was

contained into the fresh EPF approximately 7.33%. The second peak started from 200 to 320°C with maxima at 295°C. It should be due to the dehydroxylation of C-OH group from the EPF. This corresponded to 46.45%. The last peak was assigned to the combustion of remaining carbon species, contributed to 37.89% weight loss. The remaining material was balanced to be 8.33%, referred to ash. To compare the drying conditions at 60 and 100°C, OPF also was decomposed under air, and the results of TGA and TPD were presented in the same figure. The profiles for both drying conditions were found to be similar to each other. It indicated that there was no significant effect throughout the pretreatment process. Either condition could be used for the further study.

#### 2. Modified Fibers

Weight loss profiles and TPD spectrum through 550°C for EPF, mercerized fiber (MPF), and silylated fiber (SPF) were obtained from TGA and shown in Figure 2.

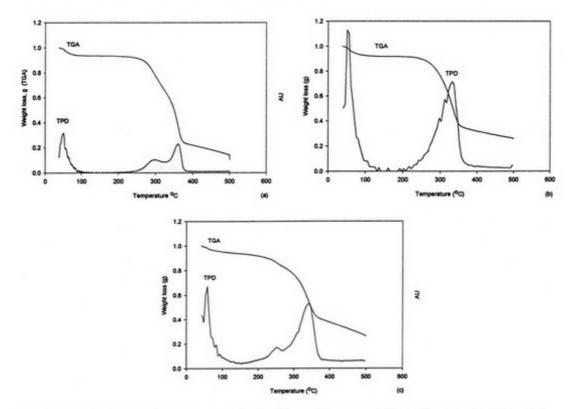


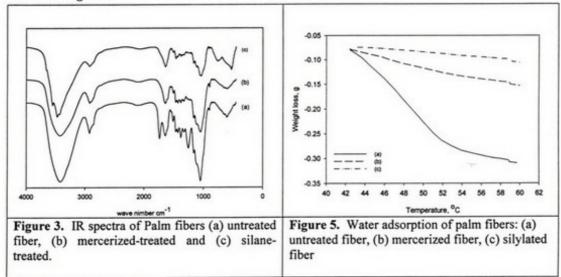
Figure 2. Weight loss and TPD spectra of palm fibers: (a) untreated fiber, (b) mercerized fiber, (c) silylated fiber

Weight loss of fiber after treatment seemed to be different from fresh fiber at the second and third steps. On the same basis, fiber after treatment contained less volatile compounds decomposed at 200-350°C. The volatile compounds could be calculated for EPF, MPF, and SPF as 84.34 to 69.72, 81.05%, respectively. However, weight loss still showed the tendency downwards after 350°C, which indicated that chemical composition containing in fiber was modified through the chemical reactions. As presenting in Figure 2a, b, and c, compositions with stronger interaction (the third step) was increased while less interaction (the second step) became reduced by the substitution reaction.

Reaction 1 Fiber-OH + NaOH → Fiber-O-Na<sup>+</sup> + H<sub>2</sub>O Reaction 2 Fiber-O-Na<sup>+</sup> + C<sub>3</sub>H<sub>9</sub>SiCl → Fiber-O-Si-C<sub>3</sub>H<sub>9</sub> + NaCl

Besides, it was found two main peaks over the TD spectrum, clearly disclosed at 40-100°C, and 200-360°C. It confirmed some amount of chemical species was leached out.

2.1FTIR: Functional groups of EPF modified through the reactions were clarified by FTIR spectrum shown in Figure 3.



Major changes are observed in the IR absorbance of alkali-treated, acetylated and silane-treated sample. Major changes are observed in the IR absorbance of mercerized-treated, and silane-treated sample. Peak at 1248 and 1736 cm<sup>-1</sup> (C-O stretching and C=O stretching, respectively) are present in the untreated natural palm fiber. On modification, these peaks disappear. This may be due to the removal of the carboxylic group by mercerizing. The carboxylic group may be present on the fiber surface from traces of fatty acids present. Peak at 2922 cm<sup>-1</sup> (C-H stretching) decreased upon the treatment.

2.2 SEM: The SEM photographs of EPF materials (Figure 4) exhibit the morphology of fibers for both fresh and treated materials.

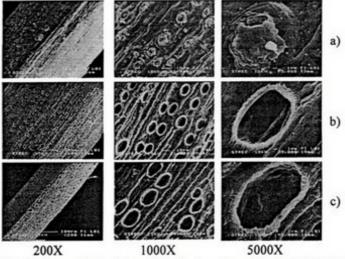


Figure 4 SEM Images of Palm Fibers both fresh and modified; a) EFP,b) Mercirized, and c)Silylated

Different magnifications of 200X, 1000X, and 5000X were shown for the development of modifications. The images of EPF presented stomata, microscopic pores in the epidermal tissue, were spread over the fiber surface. Stomata were removed after modified by mercerization and silylation. The structure became open and clearly porous. It was believed that more bounding could be formed

after pore removal. The pores were found to have an average diameter of 10, 21μm, for mercerization and silylation respectively. The porous morphology is useful for better mechanical interlocking with the matrix resin for composite fabrication [10].

Besides FTIR and SEM, materials, both fresh and treated, contain small number of surface area, according to BET surface area analysis. It indicated that adsorption would take place upon functional groups rather than physical surface adsorption.

#### 3. Hydrophobicity and adsorption measurement

Test of water adsorption: Figure 5 shows the weight loss along the heating system ranged 40-60°C of untreated palm fiber, mercerized fiber, and silylated fibers. The adsorption system was evaluated at this range since the further applications would be operated at normal to low temperature rather than high temperature, therefore, condition at 100 or 500°C would be ignored for the adsorption concept. It was found that the water contained after the adsorption on EPF much more than other modified fibers, as shown by the slope of weight loss through temperature program. The modification by mercerization and silylation could reduce water adsorption capability by 23.1 and 31.1 %, respectively. This would deal to hydroxyl groups (-OH) over fiber surface was partially removed. Hexane was selected as an absorbate for the further the hydrophobicity index (HI) calculation. Modifications by M and S could reduce the Hydrophobicity of EPF by 0.132, and 0.616.

Table 1. Sorption capacity of fibers<sup>a</sup>

Sorbent	Diesel (g/g)	HI
EPF	0.95	0.195
MPF	0.82	0.132
SPF	0.47 pate per gram of fiber	0.616

#### Conclusion

Pretreatment process, drying conditions at 60 and 100°C, was no significant effect to physical properties of the modified material. The adsorption would take place upon functional groups rather than physical surface adsorption and water adsorption on silylated fiber less than its original material. Moreover the modification by mercerization and silylation was removed to some extent of hydroxyl groups (-OH) over fiber surface that lead to reduce water adsorption capability.

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