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APPENDICES

Appendix A Calibration Curves

Table A1 Calibration curve for hydrogen (H_2)

Volume of hydrogen (ml)	Peak area
0.02	16,313
0.04	58,770
0.08	180,674
0.1	226,743
0.2	427,198
0.4	778,509

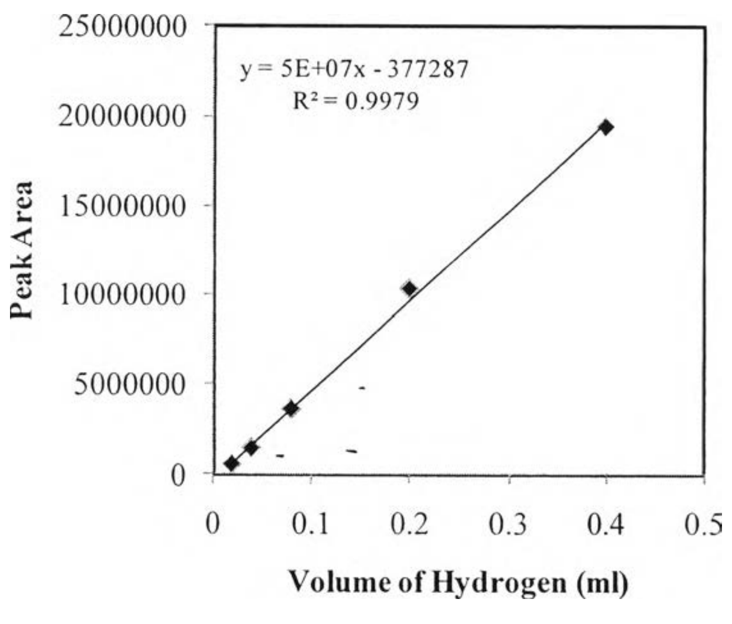

Figure A1 The relationship between volume of hydrogen (H_2) and peak area.

Equation

$$\text{Amount of hydrogen} = \frac{\text{Peak area} + 377287}{5 \times 10^7}$$

Table A2 Calibration curve for nitrogen

Volume of nitrogen (ml)	Peak area
0.02	34,210
0.04	128,767
0.08	305,287
0.1	393,916
0.2	809,433
0.4	1,602,475

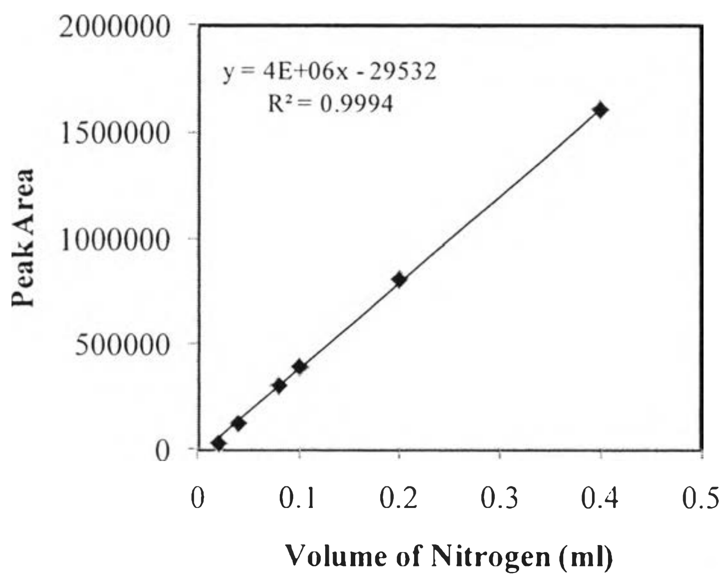

Figure A2 The relationship between volume of nitrogen (N_2) and peak area.

Equation

$$\text{Amount of nitrogen} = \frac{\text{Peak area} + 29532}{4 \times 10^6}$$

Table A3 Calibration curve for oxygen

Volume of oxygen (ml)	Peak area
0.02	12,286
0.04	43,995
0.08	104,342
0.1	135,546
0.2	280.220
0.4	562,001

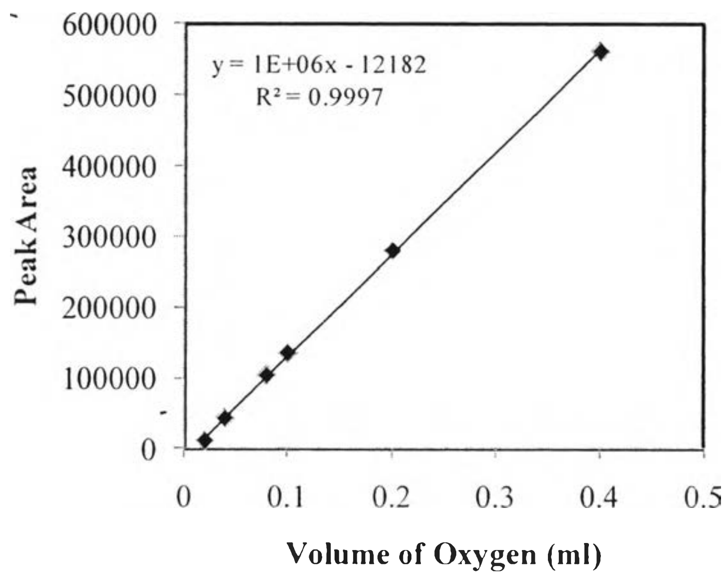

Figure A3 The relationship between volume of oxygen (O_2) and peak area.

Equation

$$\text{Amount of oxygen} = \frac{\text{Peak area} + 12182}{1 \times 10^6}$$

Table A4 Calibration curve for methane (CH_4)

Volume of methane (ml)	Peak area
0.02	92,517
0.04	381,106
0.1	1,293,552
0.2	2,674,654

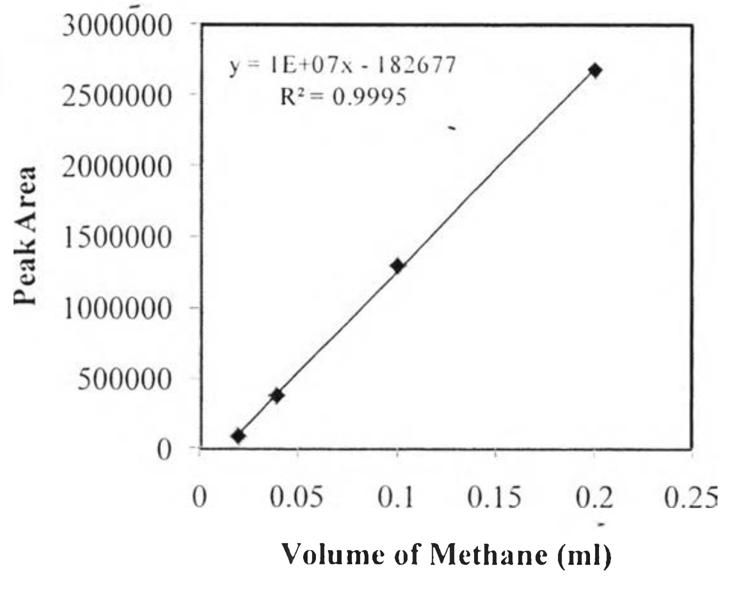

Figure A4 The relationship between volume of methane (CH_4) and peak area.

Equation

$$\text{Amount of methane} = \frac{\text{Peak area} + 182677}{1 \times 10^7}$$

Table A5 Calibration curve for carbon dioxide (CO_2)

Volume of carbon dioxide (ml)	Peak area
0.02	26,118
0.04	97,539
0.08	220,122
0.2	596,414
0.4	1,315,885

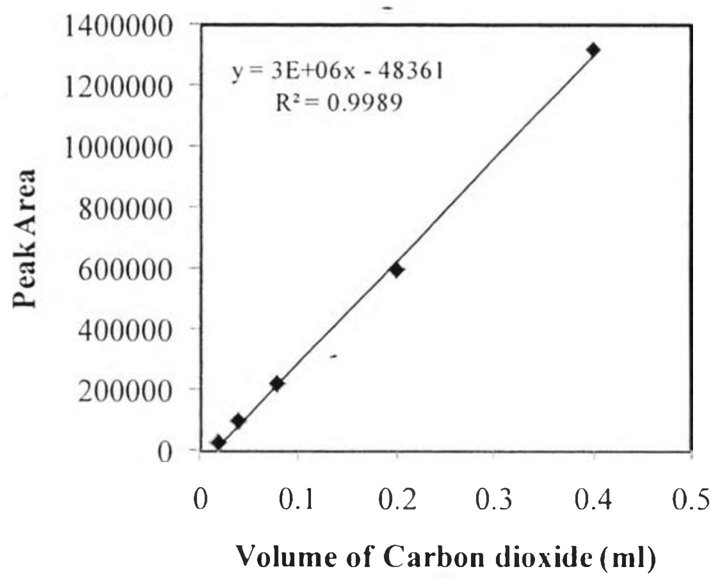

Figure A5 The relationship between volume of carbon dioxide (CO_2) and peak area.

Equation

$$\text{Amount of carbon dioxide} = \frac{\text{Peak area} + 48361}{3 \times 10^6}$$

Table A6 Calibration curve for acetic acid (CH_3COOH)

Volume of hydrogen (ml)	Peak area
1,000	0.04
2,000	0.15
3,000	0.29
4,000	0.37
5,000	0.48

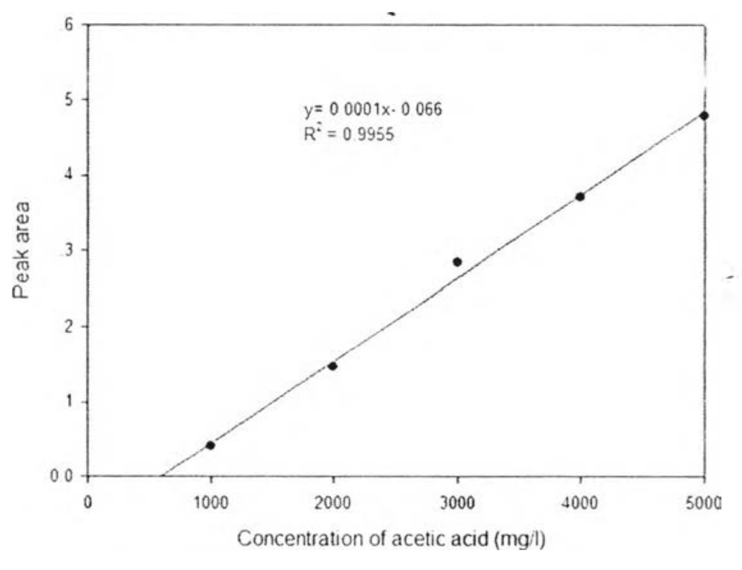

Figure A6 The relationship between concentration of acetic acid (CH_3COOH) and peak area.

Equation

$$Amount\ of\ acetic\ acid = \frac{Peak\ area + 0.066}{0.0001}$$

Table A7 Calibration curve for propionic acid (CH_3CH_2COOH)

Volume of hydrogen (ml)	Peak area
1,000	0.14
2,000	0.36
3,000	0.59
4,000	0.77
5,000	0.95

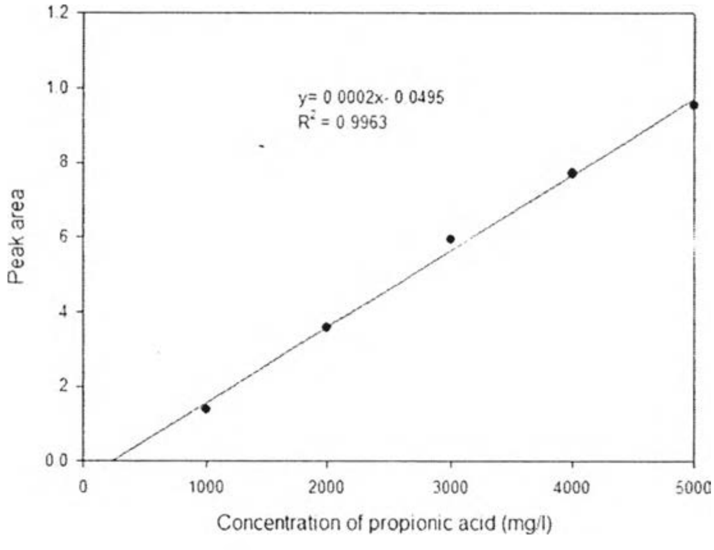

Figure A7 The relationship between concentration of propionic acid (CH_3CH_2COOH) and peak area.

Equation

$$Amount\ of\ propionic\ acid = \frac{Peak\ area + 0.0495}{0.0002}$$

Table A8 Calibration curve for butyric acid ($CH_3(CH_2)_2COOH$)

Volume of hydrogen (ml)	Peak area
1,000	0.23
2,000	0.48
3,000	0.83
4,000	1.11
5,000	1.31

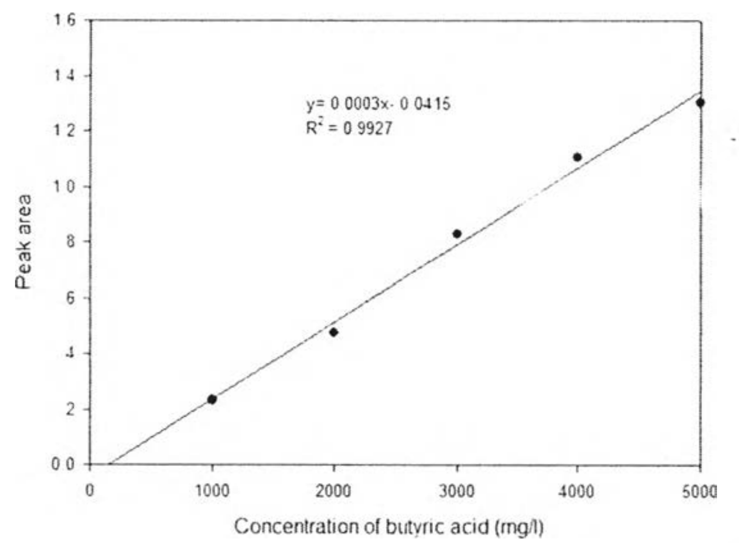

Figure A8 The relationship between concentration of butyric acid ($CH_3(CH_2)_2COOH$) and peak area.

Equation

$$Amount\ of\ butyric\ acid = \frac{Peak\ area + 0.0415}{0.0003}$$

Table A9 Calibration curve for valeric acid ($CH_3(CH_2)_3COOH$)

Volume of hydrogen (ml)	Peak area
1,000	0.21
2,000	0.51
3,000	0.80
4,000	1.19
5,000	1.36

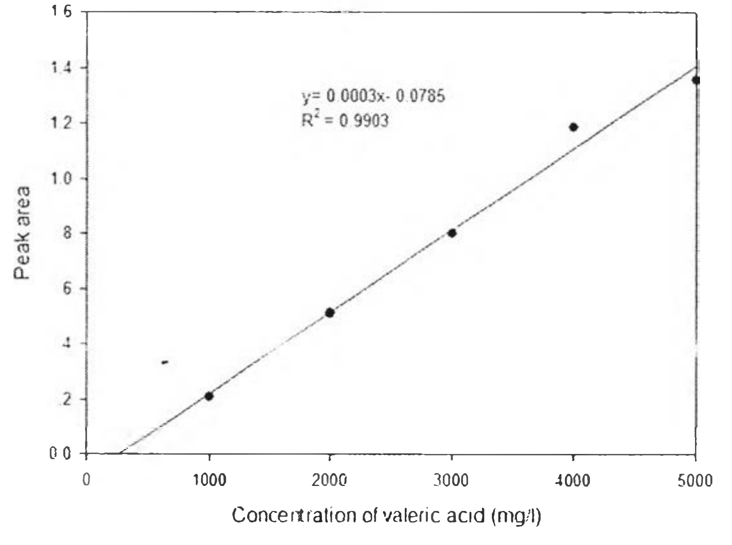

Figure A9 The relationship between concentration of valeric acid ($CH_3(CH_2)_3COOH$) and peak area.

Equation

$$Amount\ of\ valeric\ acid = \frac{Peak\ area + 0.0785}{0.0003}$$

Table A10 Calibration curve for ethanol (C_2H_5OH)

Volume of hydrogen (ml)	Peak area
1,000	0.21
2,000	0.53
3,000	0.78
4,000	1.05
5,000	1.35

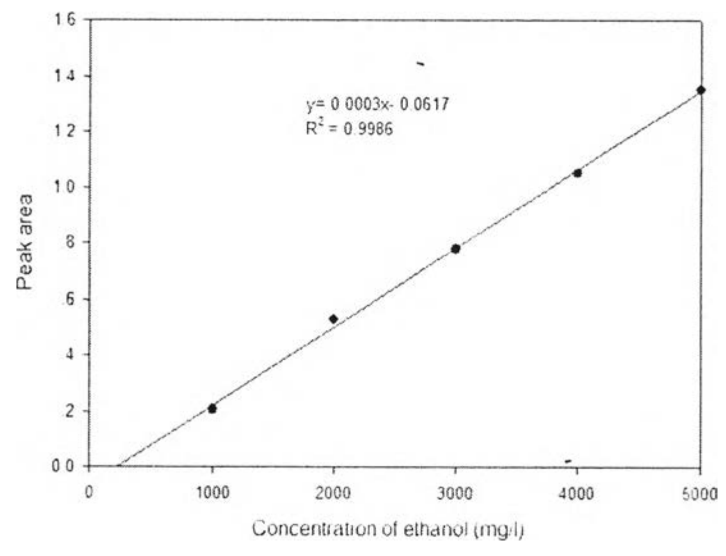

Figure A9 The relationship between concentration of ethanol (C_2H_5OH) and peak area.

Equation

$$Amount\ of\ ethanol = \frac{Peak\ area + 0.0617}{0.0003}$$

Appendix B Preparation of 5 wt./vol.% NaOH Solution for pH-controlled System

Preparation of NaOH at concentration of 5 wt./vol.%

$$= \frac{5}{100}\,\frac{g}{ml} = 50\,\frac{g}{l}$$

Appendix C Volatile Fatty Acids (VFA) Quantification by Distillation Method

C 1. Acetic Acids Stock Solution Preparation for Recovery Factor (f) Determination

Concentration of fresh acetic acid (liquid) = 99.7%

Density of acetic acid = 1.07 g/ml

Molecular weight of acetic acid = 60

Determination of fresh acetic acids concentration in term of molar

$$= \frac{0.997 \text{ L of acetic acid}}{\text{L of solution}} \times \frac{1.07 \text{ g of acetic acid}}{\text{mL of acetic acid}} \times \frac{1 \text{ mol of acetic acid}}{60 \text{ g of acetic acid}}$$

= 17.78 M

Preparation of acetic acid at concentration of 2,000 mg/L

$$= 2{,}000 \frac{\text{mg of acetic acid}}{\text{L of solution}} \times \frac{1 \text{ mole of acetic acid}}{60 \text{ g of acetic acid}}$$

= 0.0333 M

Dilution of acetic acid

$N_1V_1 = N_2V_2$

$V_1 = N_2V_2/N_1$

$= (0.0333 \times 1)/17.78$

$= 1.873 \times 10^{-3}$ L

C 2. Standard Sodium Hydroxide (0.1 M) Preparation

Concentration of fresh NaOH (solid) = 99%

Molecular weight of acetic acid = 40

Preparation of acetic acid at concentration of 0.1 M

$$= \frac{0.1 \text{ mol}}{1 \text{ L}} \times \frac{40 \text{ g}}{1 \text{ mol}} \times \frac{100}{99}$$

= 4.04 g

C 3. Recovery Factor (f) Determination

Distill 150 ml of 0.0333 M of acetic acid in distillation apparatus

Calculate the recovery factor

$$f = \frac{a}{b}$$

where

a = volatile acid concentration recovered in distillate, mg/L

b = volatile acid concentration in standard solution used, mg/L

- Find volatile acid concentration recovered in distillate by titration with 0.1 M of NaOH (MW of acetic acid = 60.5)

1) Distillate 50 ml NaOH 11.7 ml

Used NaOH	=	$11.7 \times 10^{-3} \times 0.1$
	=	1.17×10^{-3} mol
Acetic acid in distillate	=	1.17×10^{-3} mol
	=	$1.17 \times 10^{-3} \times 60.5$
	=	0.07 g
Concentration of acetic acid in distillate		
	=	0.07/50
	=	1.405×10^{-3} g/ml
	=	1,405 mg/l

2) Distillate 25 ml NaOH 5.7 ml

Used NaOH	=	$5.7 \times 10^{-3} \times 0.1$
	=	5.7×10^{-4} mol
Acetic acid in distillate	=	5.7×10^{-4} mol
	=	$5.7 \times 10^{-4} \times 60.5$
	=	0.034 g
Concentration of acetic acid in distillate		
	=	0.034/25
	=	1.368×10^{-3} g/ml
	=	1,368 mg/l
Average	=	1,387 mg/l
Recovery factor (f)	=	1,387/2,000
	=	0.693

CURRICULUM VITAE

Name: Ms. Patcharee Intanoo

Date of Birth: July 6, 1985

Nationality: Thai

University Education:

2004–2008 Bachelor Degree of Industrial Chemistry, Faculty of Science, Chiang Mai University, Chiang Mai, Thailand

2008-2010 Master Degree of Polymer Science, The Petroleum and Petrochemical College, Chulalongkorn University, Bangkok, Thailand

Work Experience

March-May 06	Position:	Quality Control
	Company name:	Bangkok Polyethylene Co., Ltd., Maptaphut Rayong, Thailand

Publication:

1. Intanoo, P;. Rangsunvigit, P.; Namprohm, W.; Thamprajamchit, B.; Chavadej, J.; and Chavadej, S. (2012), Hydrogen production from alcohol wastewater by an anaerobic sequencing batch reactor under thermophilic operation: Nitrogen and phosphorous uptakes and transformation, International Journal of Hydrogen Energy, 37, 11104-11112.
2. Intanoo, P.; Suttikul, T.; Leethochawalit, M.; Gulari, E,; and Chavadej, S. (2014), Hydrogen Production from Alcohol Wastewater with Added Fermentation Residue by an Anaerobic Sequencing Batch Reactor (ASBR) under Thermophilic Operation, International Journal of Hydrogen Energy, accepted, in press.

Proceeding:

1. Intanoo, P.; Chavadej, J.; and Chavadej, S. (2012, April 11-13) Effect of COD Loading Rate on Hydrogen Production from Alcohol Wastewater. Proceeding of the ICCBEE 2012: International Conference on Chemical, Biological and Environmental Engineering, Venice, Italy.

Presentations:

1. Intanoo, P.; Chavadej, J.; and Chavadej, S. (2012, April 11-13) Effect of COD Loading Rate on Hydrogen Production from Alcohol Wastewater. Paper presented at ICCBEE 2012: International Conference on Chemical, Biological and Environmental Engineering, Venice, Italy.
2. Intanoo, P.; Chavadej, S.; and Rangsunvigit, P. (2012, November 11-13) Hydrogen Production from Alcohol Wastewater with Cellulosic Residue by an Anaerobic Sequencing Batch Reactor (ASBR) under Thermophilic Operation. Paper presented at The First International Symposium on Advanced Water Science and Technology, Noyori Conference Hall, Nagoya University, Nagoya Japan.