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APPENDICES

Appendix A

1. Proximate analysis of biomass

1.1 Moisture analysis: ASTM D3173

Moisture is determined by establishing the loss in weight of the sample when heated under rigidly controlled conditions of temperature, time and atmosphere, sample weight, and equipment specifications.

Procedure

1. Dry sample container for 30 min at $103 \pm 1^\circ\text{C}$ in the oven, then cool in desiccator to room temperature. Weigh to the nearest 0.02 g and record as container weight, W_c . Place a minimum of 1 g of sample in the container, weigh the sample and container to the nearest 0.01 g and record as initial weight, W_i .
2. Place the sample and container in the oven for 16 h at $103 \pm 1^\circ\text{C}$.
3. Remove the sample and the container from the oven and cool in the desiccator to room temperature. Remove the sample and container from the desiccator, weigh immediately to the nearest 0.01 g, and record the weight.
4. Return the sample and container to the oven at $103 \pm 1^\circ\text{C}$ for 2 h. Repeat.
5. Continue 4 until the total weight change between weighing varies less than 0.02 % and record as the final weight, W_f .

Calculation

$$\text{Moisture in analysis sample, (\%)} = [(W_i - W_f) / (W_i - W_c)] \times 100$$

Where : W_c = container weight, g.

W_i = initial weight, g. and

W_f = final weight, g.

1.2 Volatile matter analysis: ASTM D3175

Volatile matter is determined by establishing the loss in weight resulting from heating wood under rigidly controlled conditions.

Procedure

1. Weigh the crucible and cover to the nearest 0.01 g and record as crucible weight, W_c . Place approximately 1 g of sample in the crucible, cover, weigh the crucible, cover, weigh the crucible, cover, and sample to the nearest 0.01 g, and record as initial weight, W_i .
2. Place the covered crucible with sample on platinum or nickel-chromium wire supports and insert directly into the furnace chamber, which shall be maintained at a temperature of 950°C , and lower immediately to the 950°C .
3. Raising the crucible to the top of the furnace chamber, then lower immediately back to the 950°C . After heating for a total of exactly 7 min, remove the crucible from the furnace, cool in a desiccator.
4. Weigh the covered crucible with sample as soon as cold to the nearest 0.1 mg and record as final weight, W_f .

Calculation

1. Calculate the weight loss percent as follows :

$$\text{Weight loss, \%} = 100 \times (W_i - W_f) / (W_i - W_c) = A \quad (1)$$

Where: W_c = weight of crucible and cover, g,

W_i = initial weight, g, and

W_f = final weight, g.

2. Calculate the volatile matter percent in the analysis samples as follows:

$$\text{Volatile matter in analysis sample, \%} = A - B \quad (2)$$

Where: A = weight loss %, and

B = moisture, %.

1.3 Ash analysis: ASTM D3174

The ash content is an approximate measure of the mineral content and other inorganic matter in wood.

Procedure

1. Ignite the empty crucible and cover over a burner or in the muffle at 600, cool in a desiccator, and weigh to the nearest 0.1 mg. Place the 2-g test specimen in the crucible, determine the weight of crucible plus specimen, and place in the drying oven at 100 to 105 with the crucible cover removed. After 1 h, replace the cover on the crucible, cool in a desiccator, and weigh. Repeat the drying and weighing until the weight is constant.

2. Place the crucible and contents, with the cover removed, in the muffle furnace and ignite until all the carbon is eliminated. Heat slowly at the start to avoid flaming and protect the crucible from strong drafts at all times to avoid mechanical loss of test specimen. The recommended temperature of final ignition is 580 to 600 °C. Avoid heating above this maximum.

3. Remove the crucible with its contents to a desiccator, replace the cover loosely, cool, and weigh accurately. Repeat the heating for 30-min periods until the weight after cooling is constant to within 0.2 mg.

Calculation

Calculate the percentage of ash, based on the weight of the moisture-free wood, as follows:

$$\text{Ash, \%} = (W_1 / W_2) \times 100$$

Where:

W_1 = weight of ash, and

W_2 = weight of oven-dry sample.

Appendix B

Calculation of gas composition

Peak area of standard gas from gas chromatography

std	No. 1 area	No. 2 area	Average area	% balance in He
H ₂	838257.8	881719.7	859988.8	75
CO	274.4	167.2	220.8	10
CH ₄	53899.1	51486	52692.55	10
CO ₂	20304.7	23021.4	21663.05	5

Calculation of gas products from experiment;

1. Calculation gas composition (%)

$$\% \text{H}_2 = \frac{(75/100) \times [\text{Peak area of H}_2 \text{ from experiment}]}{[\text{Peak area of H}_2 \text{ Standard gas}]}$$

$$\% \text{CO} = \frac{(10/100) \times [\text{Peak area of CO from experiment}]}{[\text{Peak area of CO Standard gas}]}$$

$$\% \text{CH}_4 = \frac{(10/100) \times [\text{Peak area of CH}_4 \text{ from experiment}]}{[\text{Peak area of CH}_4 \text{ Standard gas}]}$$

$$\% \text{CO}_2 = \frac{(5/100) \times [\text{Peak area CO}_2 \text{ from experiment}]}{[\text{Peak area of CO}_2 \text{ Standard gas}]}$$

2. Carbon in feed

$$\text{Carbon in coconut} = \frac{\text{Carbon from elemental analysis (\%)} \times \text{biomass feed rate}}{12}$$

12

3. Calculation carbon of gas product

$$\text{CO} = \frac{\text{flow out (ml/min)} \times (\% \text{CO}) \times 298}{273 \times 22.4} \text{ mmol/min}$$

$$\text{CH}_4 = \frac{\text{flow out (ml/min)} \times (\% \text{CO}) \times 298}{273 \times 22.4} \text{ mmol/min}$$

$$\text{CO}_2 = \frac{\text{flow out (ml/min)} \times (\% \text{CO}) \times 298}{273 \times 22.4} \text{ mmol/min}$$

$$4. \text{ Carbon conversion (\%)} = \frac{\text{CO} + \text{CH}_4 + \text{CO}_2}{\text{Carbon total in feed}} \times 100$$

$$\text{Carbon char (\%)} = \frac{\text{carbon from elemental analysis (\%)} \times \text{char weight (g)}}{\text{Carbon total in feed}} \times 100$$

$$\text{Tar (\%)} = 100 - \text{gas (\%)} - \text{char (\%)}$$

$$5. \text{ Steam to carbon ratio (S/C)} = \frac{\text{steam feed rate (g/min)}}{\text{Carbon total in feed} \times 18}$$

Appendix C

Experimental Data

Data		Gas composition (%)			
		H ₂	CO	CH ₄	CO ₂
Calcination	500	35.39	21.12	17.02	26.22
temperature	750	38.42	22.70	10.84	18.01
	950	22.99	15.21	25.83	35.96
coconut	noncat	22.67	32.31	15.11	29.90
	catalyst	30.31	37.71	4.74	27.22
plam	noncat	22.42	32.70	16.85	28.02
	catalyst	33.94	34.02	6.91	5.11
temperature	650	30.89	35.56	13.37	20.15
coconut	700	35.68	29.82	12.80	21.65
	750	42.34	29.52	10.84	17.30
	800	45.00	32.00	8.01	15.09
plam	650	11.74	48.87	18.50	20.89
	700	30.89	35.59	13.37	20.15
	750	35.68	29.82	12.85	21.65
	800	42.34	29.52	10.84	17.30
Steam/carbon	0.24	41.67	27.36	15.33	15.40
coconut	0.44	42.94	20.17	15.22	17.65
	0.95	46.11	20.34	11.52	22.02
plam	0.24	41.67	27.36	15.33	15.63
	0.44	40.94	26.17	15.22	17.65
	0.95	49.11	17.34	11.52	22.02
oxygen	10	34.73	64.76	0.01	0.49
plam	20	17.88	79.82	0.06	2.22
	30	17.47	77.36	0.01	5.15
	40	9.93	78.86	0.01	11.18
oxygen	10	30.42	60.66	0.71	8.20
plam	20	28.13	70.81	77.36	78.86
	30	17.47	77.36	0.02	5.15
	40	10.00	78.86	0.01	11.18

Appendix D

Data from gas chromatography

Condition	Gas composition	No 1 (area)	No. 2 (area)	Avg (area)
palm shell non catalyst 700 °C (S/C) 0.24	H ₂	145401.6	145401.6	145401.6
	CO	150.8	150.8	150.8
	CH ₄	81128.4	81128.4	81128.4
	CO ₂	57827.9	57827.9	57827.9
coconut shell non catalyst 700 °C (S/C) 0.24	H ₂	90968.8	89696.9	90332.85
	CO	348.6	364.2	356.4
	CH ₄	57089.7	56153.5	56621.6
	CO ₂	51610.3	51761.4	51685.85
palm shell 700 °C (S/C) 0.24 Ni/dolomite 10 g.	H ₂	86354.5	88866.1	87610.3
	CO	691.7	399.9	545.8
	CH ₄	57858.1	51396.5	54627.3
	CO ₂	57858.1	56091.5	56974.8
coconut shell 700 °C (S/C) 0.24 Ni/dolomite 10 g.	H ₂	88996.4	87207.3	88101.85
	CO	1428.8	1321.3	1375.05
	CH ₄	51904.4	51033.8	51469.1
	CO ₂	51904.4	60529	56216.7
palm shell 650 °C (S/C) 0.24 Ni/dolomite 10 g.	H ₂	54458	34985.3	44721.65
	CO	186	2570.3	1378.15
	CH ₄	59056.2	59056.2	59056.2
	CO ₂	35798	35798	35798
palm shell 700 °C (S/C) 0.24 Ni/dolomite 10 g.	H ₂	178669.5	122094.2	150381.9
	CO	75.5	2489.6	1282.55
	CH ₄	63627.9	45527.5	54577.7
	CO ₂	46137.6	42117.7	44127.65
palm shell 750 °C (S/C) 0.24 Ni/dolomite 10 g.	H ₂	20117.89	19948.19	20033.04
	CO	6283.67	6653.50	6468.59
	CH ₄	14790.18	14690.71	14740.45
	CO ₂	13733.36	13764.83	13749.10
palm shell 800 °C (S/C) 0.24 Ni/dolomite 10 g.	H ₂	297069.1	262341.5	279705.3
	CO	1444	1444	1444
	CH ₄	51352	68746.4	60049.2
	CO ₂	51352.8	51506.9	51429.85

Condition	Gas composition	No. 1 (area)	No 2 (area)	Avg (area)
coconut shell	H ₂	56587.5	26534.3	41560.9
650 °C (S/C) 0.24	CO	1771.1	4311.9	3041.5
Ni/dolomite 10 g.	CH ₄	21245.7	11050.8	16148.25
	CO ₂	38337.9	24589.3	31463.6
coconut shell	H ₂	50374.9	11169.8	30772.35
700 °C (S/C) 0.24	CO	955	6464.6	3709.8
Ni/dolomite 10 g.	CH ₄	31826.4	3493.7	17660.05
	CO ₂	43297.5	5461.3	24379.4
coconut shell	H ₂	54906	54906	54906
750 °C (S/C) 0.24	CO	1558.1	1558.1	1558.1
Ni/dolomite 10 g.	CH ₄	36496	36496	36496
	CO ₂	36570.8	36570.8	36570.8
coconut shell	H ₂	83960.8	52753.1	68356.95
800 °C (S/C) 0.24	CO	1309	2808.8	2058.9
Ni/dolomite 10 g.	CH ₄	46799.5	34572	40685.75
	CO ₂	48046.9	36732.6	42389.75
palm shell	H ₂	180210.6	101421.8	140816.2
800 °C (S/C) 0.24	CO	1706	1406	1556
Ni/dolomite 10 g.	CH ₄	37125.4	37125.4	37125.4
	CO ₂	54249	37146.4	45697.7
palm shell	H ₂	203737.9	68482.6	136110.3
800 °C (S/C) 0.44	CO	878	4682.5	2780.25
Ni/dolomite 10 g.	CH ₄	64825.4	27203.8	46014.6
	CO ₂	46340	25799.2	36069.6
palm shell	H ₂	303225.6	273096.5	288161.1
800 °C (S/C) 0.95	CO	645.1	609.1	627.1
Ni/dolomite 10 g.	CH ₄	69171.9	65088.3	67130.1
	CO ₂	50642.2	50642.2	50642.2
coconut shell	H ₂	64982.9	31109.2	48046.05
800 °C (S/C) 0.24	CO	2132	5490.6	3811.3
Ni/dolomite 10 g.	CH ₄	50423.2	13106.4	31764.8
	CO ₂	46440.7	12816.2	29628.45
coconut shell	H ₂	48833.3	46620.4	47726.85
800 °C (S/C) 0.44	CO	2495	1450.9	1972.95
Ni/dolomite 10 g.	CH ₄	43631.7	44343.8	43987.75
	CO ₂	48059.8	40067.9	44063.85
coconut shell	H ₂	106866.1	103504.1	105185.1
800 °C (S/C) 0.95	CO	1184	1754	1754
	CH ₄	64982.9	31109.2	48046.05
	CO ₂	56587.5	26534.3	41560.9

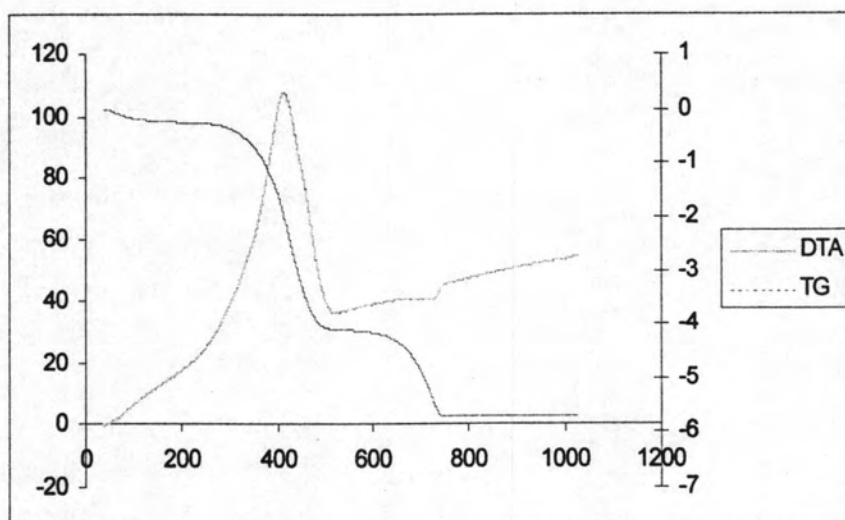
Condition	Gas composition	No. 1 (area)	No 2 (area)	Avg (area)
palm shell	H2	6322	6322	6322
800 °C (S/C) 0.95	CO	5005	5005	5005
O ₂ 10 ml	CH4	481	481	481
	CO2	11020	11020	11020
palm shell	H2	655	655	655
800 °C (S/C) 0.95	CO	1241	1241	1241
O ₂ 20 ml	CH4	481	481	481
	CO2	10063	10063	10063
palm shell	H2	359	359	359
800 °C (S/C) 0.95	CO	675	675	675
O ₂ 30 ml	CH4	58	58	58
	CO2	13069	13069	13069
palm shell	H2	135	135	135
800 °C (S/C) 0.95	CO	455	455	455
O ₂ 40 ml	CH4	48	48	48
	CO2	18766	18766	18766
coconut shell	H2	1062	1062	1062
800 °C (S/C) 0.95	CO	899	899	899
O ₂ 10 ml	CH4	5040	5040	5040
	CO2	35347	35347	35347
coconut shell	H2	477	477	477
800 °C (S/C) 0.95	CO	620	620	620
O ₂ 20 ml	CH4	3100	3100	3100
	CO2	13508	13508	13508
coconut shell	H2	359	359	359
800 °C (S/C) 0.95	CO	675	675	675
O ₂ 30 ml	CH4	58	58	58
	CO2	13069	13069	13069
coconut shell	H2	135	135	135
800 °C (S/C) 0.95	CO	455	455	455
O ₂ 40 ml	CH4	48	48	48
	CO2	18766	18766	18766
coconut shell	H2	185758.8	61153.6	123456.2
800 °C (S/C) 0.95	CO	615.5	4781.6	2698.55
Ni/Pt/Dolomite	CH4	36148.4	16603.3	26375.85
	CO2	32136.5	15768.2	23952.35
coconut shell	H2	96245.2	96245.2	96245.2
800 °C (S/C) 0.95	CO	753	753	753
Ni/Fe/Dolomite	CH4	54249.9	54249.9	54249.9
	CO2	52588.1	52588.1	52588.1

Condition	Gas composition	No. 1 (area)	No 2 (area)	Avg (area)
coconut shell 800 °C (S/C) 0.95 Ni/Co/Dolomite	H2	1307.4	1220.2	17185.3
	CO	4204.1	4269.2	4236.65
	CH4	7128.7	70329.4	38729.05
	CO2	17185.3	7128.7	12157

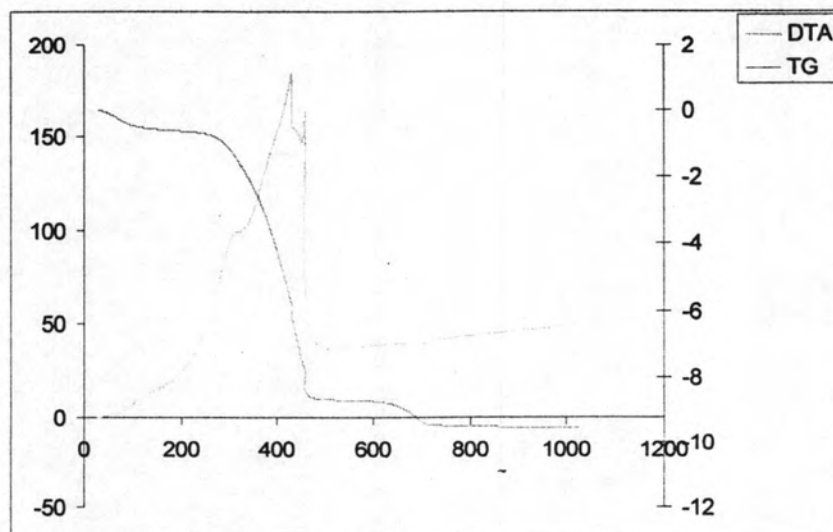
Appendix E

Data from coke formation by thermogravimetric analysis of various catalysts after reaction

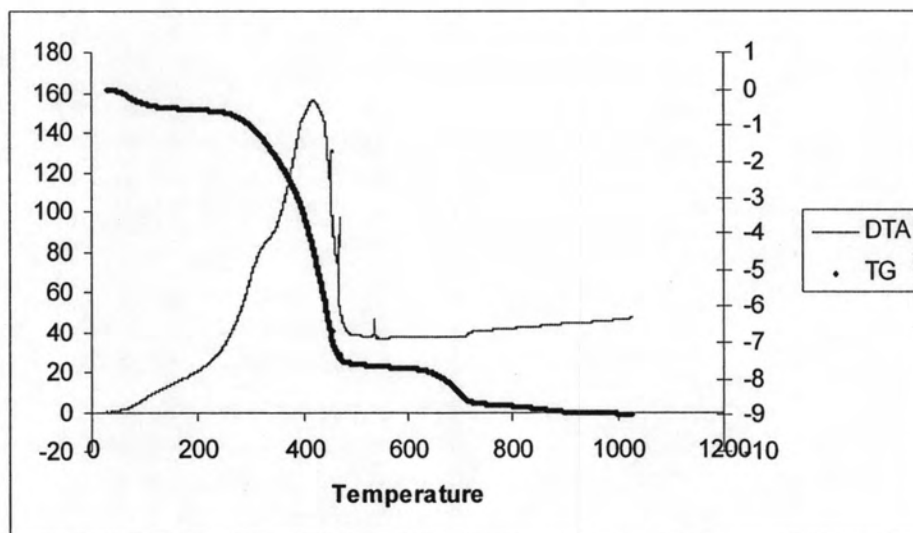
1. Used Ni



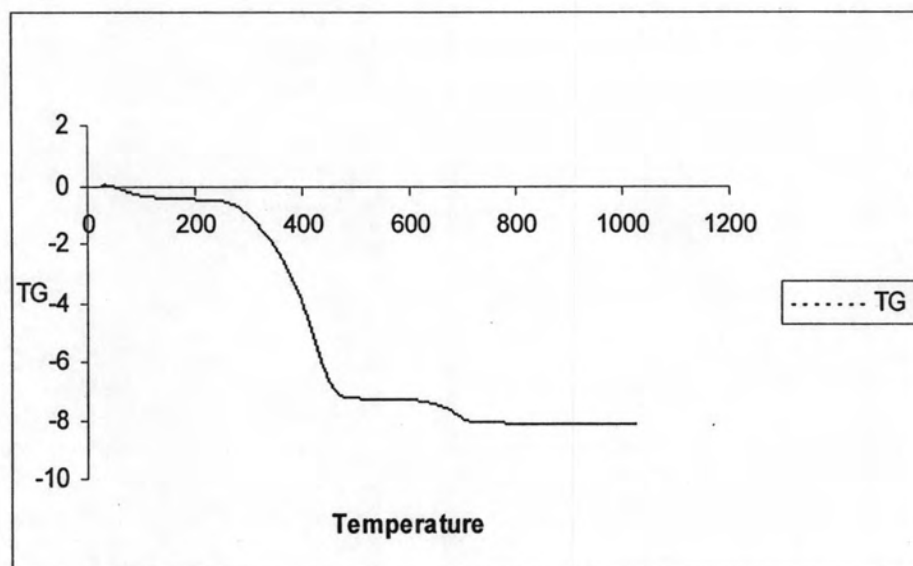
2. Used Ni/Pt/DM



3. Used Ni/Co/DM



4. Used Ni/Fe/DM



CURRICULUM VITAE

Name: Miss Pattaraporn Chaiprasert

Address: 544/33 Chan Raad, Yannawa, Bangkok 10120, Thailand

Education

2003-Present Ph.D course in department of Chemical Technology, Faculty of Science,
Chulalongkorn University, Thailand

1996-1999 Master Degree of Science in Petrochemistry and Polymer Science
Faculty of Science, Chulalongkorn University, Thailand

1993-1996 Bachelor Degree of Science in Chemistry Faculty of Science,
Mahidol University, Thailand