ภาคผนวก

ภาคผนวก ก

การคำนวณ

การคำนวณ

1. **การคำนวณความหนาของเตาปฏิกรณ์**

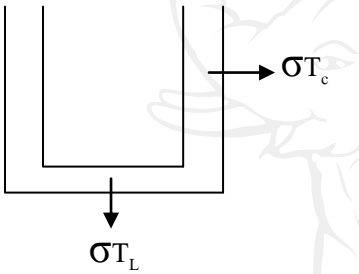
Stainless Steel 314 Tensile Strength, Ultimate = 689 MPa

Safety factor = 3

Diameter = 65 mm.

Ultimate strength = (689/3)

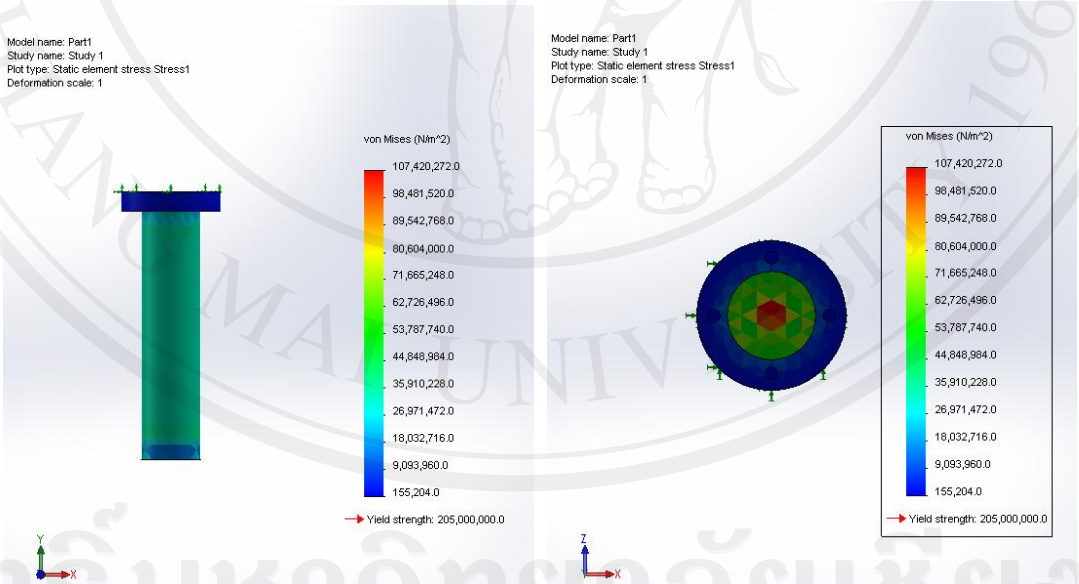
= 229.67 MPa



$$\sigma T_c = \frac{P(MPa) * ri\,(m)}{2t(m)}$$

t = (3.5 * 0.065) / (4*229.67)

= 0.875 mm.



รูป ก-1 ลักษณะ yield strength ในเตาปฏิกรณ์ขนาด 3.5 MPa

2. **การคำนวณร้อยละผลได้**

ร้อยละผลได้(%yield) = (น้ำหนักถ่านหลังการทดลอง/ น้ำหนักก่อนชีวมวลการทดลอง) *100

ตัวอย่าง ขนาดอนุภาคน้อยกว่า 3.0 มิลลิเมตร ที่สภาวะ1 ชั่วโมง, (B/C) เท่ากับ 1:0

น้ำหนักก่อนทดลอง = 19.92

น้ำหนักหลังทดลอง = 11.42

ดังนั้น %yield = (11.42/19.92)*100

= 57.32%

3. **การคำนวณหาพลังงานที่เปลี่ยนแปลงของน้ำ**

. การเปลี่ยนแปลงพลังงานเมื่ออุณหภูมิไม่คงที่

ใช้สูตร $\Delta H = ms(\Delta t)$

ΔH = พลังงานที่เปลี่ยนแปลงไป

m = มวล

s = ความร้อนจำเพาะ

Δt = อุณหภูมิที่เปลี่ยนแปลง

การเปลี่ยนแปลงพลังงานเมื่ออุณหภูมิคงที่ (ความร้อนแฝง)

ใช้สูตร $\Delta H = mL$

ΔH = พลังงานที่เปลี่ยนแปลงไป

ตัวอย่าง : ให้ความร้อนกับน้ำแข็ง 10 กรัม 0˚C จนเป็นไอน้ำ 10 กรัม 100˚Cต้องใช้พลังงานทั้งหมดเท่าใด (กำหนดค่าความร้อนจำเพาะของน้ำ =4.2 J/g˚C ; ความร้อนแฝงของการหลอมเหลวของน้ำ = 334.8 J/g ; ความร้อนแฝงของการกลายเป็นไอ = 2,256 J/g)

วิธีคิด : เราต้องทำทีละขั้นตอนดังนี้

1. น้ำแข็ง 10 กรัม 0˚C $\rightarrow$ น้ำ 10 กรัม 0˚C ต้องใช้ความร้อนแฝงการหลอมเหลว

$\Delta H = mL \rightarrow \Delta H$ = 10 X 334.8 = 3,348 J

2. น้ำ 10 กรัม 0˚C $\rightarrow$ น้ำ 10 กรัม 100˚C

$\Delta H = ms(\Delta t) \rightarrow \Delta H$ = 10 X 4.2 X (100-0) = 4,200 J

3.น้ำ 10 กรัม 100°C $\rightarrow$ ไอน้ำ 10 กรัม 100°C ต้องใช้ความร้อนแฝงการกลายเป็นไอ

$\Delta H = mL \rightarrow \Delta H$ = 10 X 2256 = 22,560 J

4. รวมพลังงานที่ต้องใช้ทั้งหมด = 3348+4200+22560 = 30,108 J

4. **การคำนวณหาค่าไฟฟ้ากระแสเข้า**

4.1 พลังงานไฟฟ้าขณะเริ่มการทอลอง (25 – 220 °C)

Temp	ประสิทธิภา(%)	time(s)	MJ
0-100	50	600	0.600
100-160	100	382	0.764
160-200	11.17	258	0.058
200-220	16.81	1210	0.407

พลังงานไฟฟ้าที่ใช้ (MJ) = (heater (2000W)*ประสิทธิภาพกาทำงาน*เวลา)/10^6

= (2000*0.5*600)/10^6

= 0.6 MJ

4.2 พลังงานไฟฟ้าขณะทดลอง (220°C)

reactor (W)	time(s)	ประสิทธิภา (%)	J	MJ
2000	3600	0.152	1094400	1.0944
2000	7200	0.152	2188800	2.1888
2000	14400	0.152	4377600	4.3776
2000	21600	0.152	6566400	6.5664

พลังงานไฟฟ้าที่ใช้ (MJ) = (heater (2000W)*ประสิทธิภาพกาทำงาน*เวลา)/10^6

= (2000*0.152*3600)/10^6

= 1.094 MJ

เวลา(h)	พลังงานไฟฟ้าที่ป้อนทั้งหมด(MJ)
1	2.923
2	4.017
4	6.206
6	8.395

5. **คำนวณการสูญเสียความร้อนจากผนัง**

5.1 สูญเสียความร้อนหน้าประเก็น

$Q = Q_{conv} + Q_{red}$

$Q = h(\pi DL)(Th-T\infty)+\varepsilon\sigma(\pi DL)(T^4h-T^4s)$

หาอัตราการถ่ายเทความร้อนต่อความยาวท่อคือ

$$\frac{Q}{L} = h(\pi D)(T_h - T_\infty) + \varepsilon\sigma(\pi D)(T_h^4 - T_s^4)$$

$$\frac{Q}{L} = 15W/m^2K(\pi \times 0.07m)(220-25)^\circ C + 0.8(5.67\times10^{-8})(\pi \times 0.07m)(493^4 - 298^4)K$$

$$\frac{Q}{L} = 1386.9W/m$$

$$Q = 69.34w$$

time	W	total (MJ)
3600	69.34	0.250
7200	69.34	0.499
14400	69.34	0.998
21600	69.34	1.498

5.2 สูญเสียความร้อนบริเวณผนังเตาปฏิกรณ์

Loss ผนัง (100^oC) = 0.37 kw/m (ตาราง ค-5)

time	kw/m	area	MJ
3600	0.37	0.23236	0.836
7200	0.37	0.23236	1.673
14400	0.37	0.23236	3.346
21600	0.37	0.23236	5.019

ค่าความร้อน (Q) = เวลา* Loss ผนัง ($100^{o}C$)*2πh

= 3600*0.37*2*3.14*0.1

= 0.836 MJ

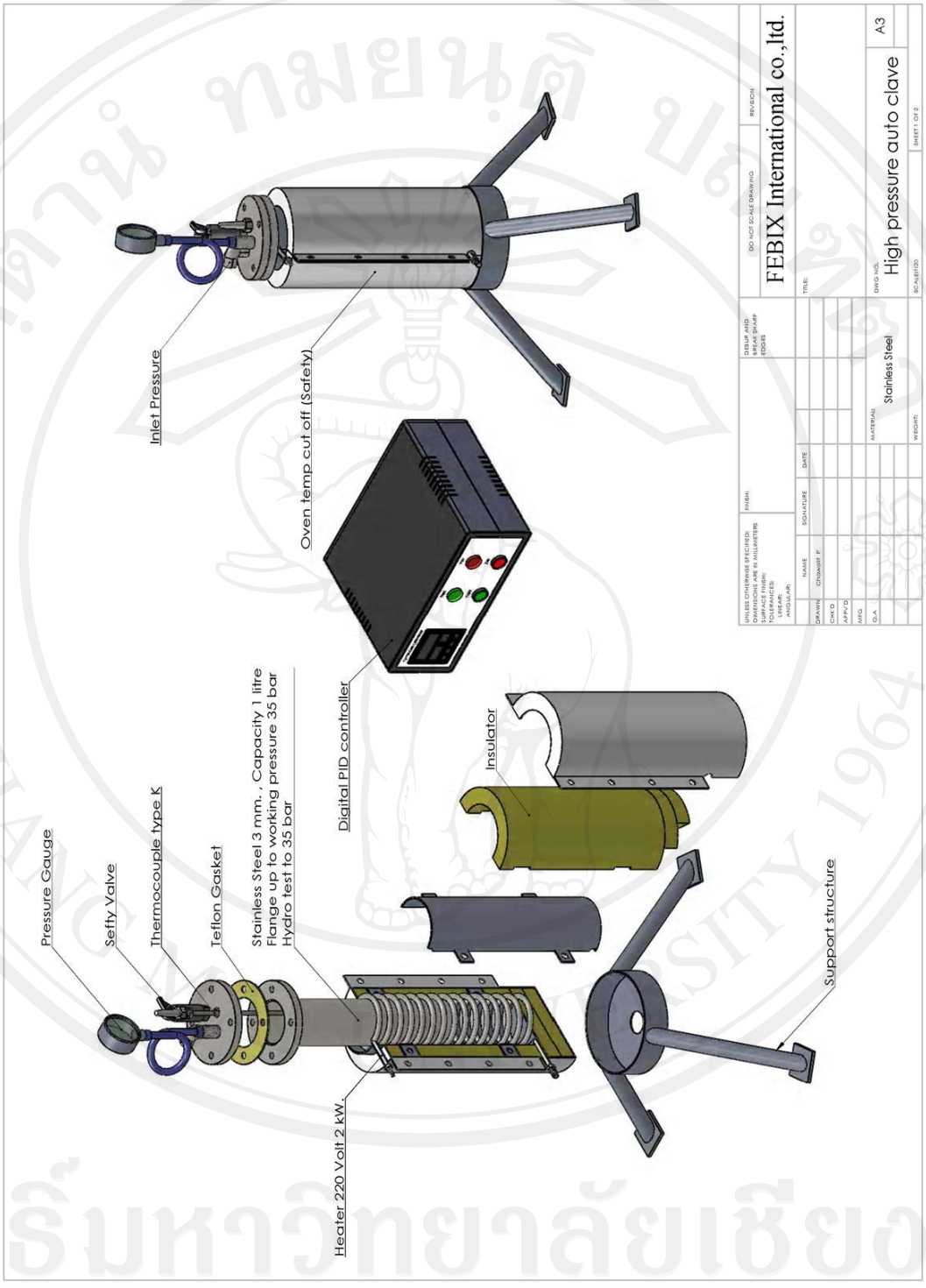
Time	Total loss
3600	1.086
7200	2.172
14400	4.344
21600	6.517

ภาคผนวก ข

ข้อมูลและการออกแบบเตาปฏิกรณ์

ตาราง ข-1 แสดงคุณสมบัติของเหล็กกล้าไร้สนิม (Stainless Steel 314)

Physical Properties	**Metric**	**English**	**Comments**
Density	7.8 g/cc	0.282 lb/in^3	
Mechanical Properties			
Hardness, Brinell	180	180	
Hardness, Knoop	201	201	Converted from Brinell Hardness
Tensile Strength, Ultimate	689 MPa	99900 psi	
Tensile Strength, Yield	345 MPa	50000 psi	
Elongation at Break	45 %	45 %	in 50 mm
Modulus of Elasticity	200 GPa	29000 ksi	
Electrical Properties			
Electrical Resistivity	7.7e-005 ohm-cm	7.7e-005 ohm-cm	at 20ºC
Magnetic Permeability	1.02	1.02	approximate value for the annealed condition at RT
Thermal Properties			
CTE, linear 250°C	15.1 µm/m-°C	8.39 µin/in-°F	at 0-315°C (32-600°F)
CTE, linear 500°C	17.6 µm/m-°C	9.78 µin/in-°F	0 - 815ºC
Specific Heat Capacity	0.5 J/g-°C	0.12 BTU/lb-°F	from 0-100°C (32-212°F)
Thermal Conductivity at Elevated Temperature	17.5 W/m-K	121 BTU-in/hr-ft^2-°F	100ºC



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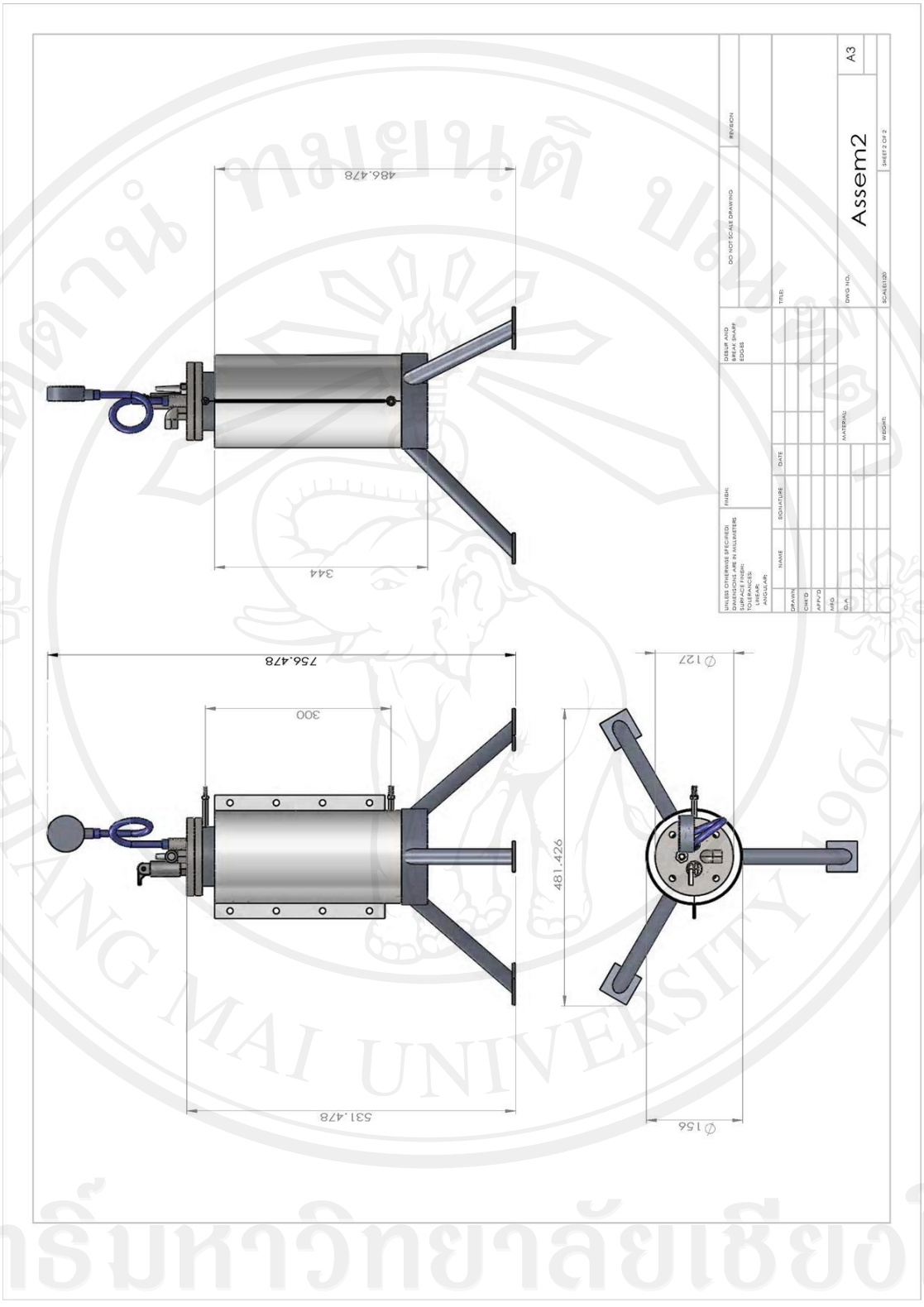

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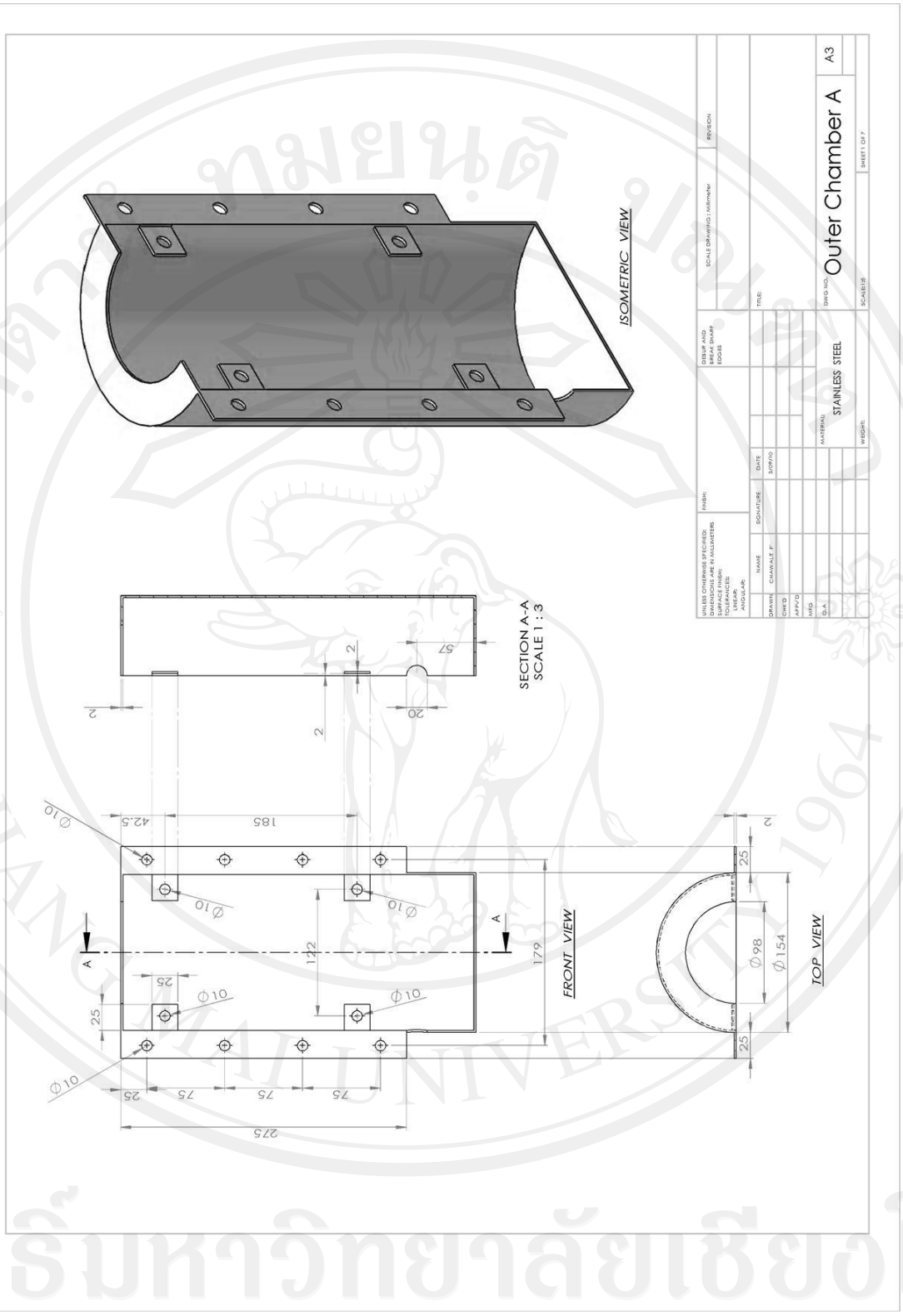

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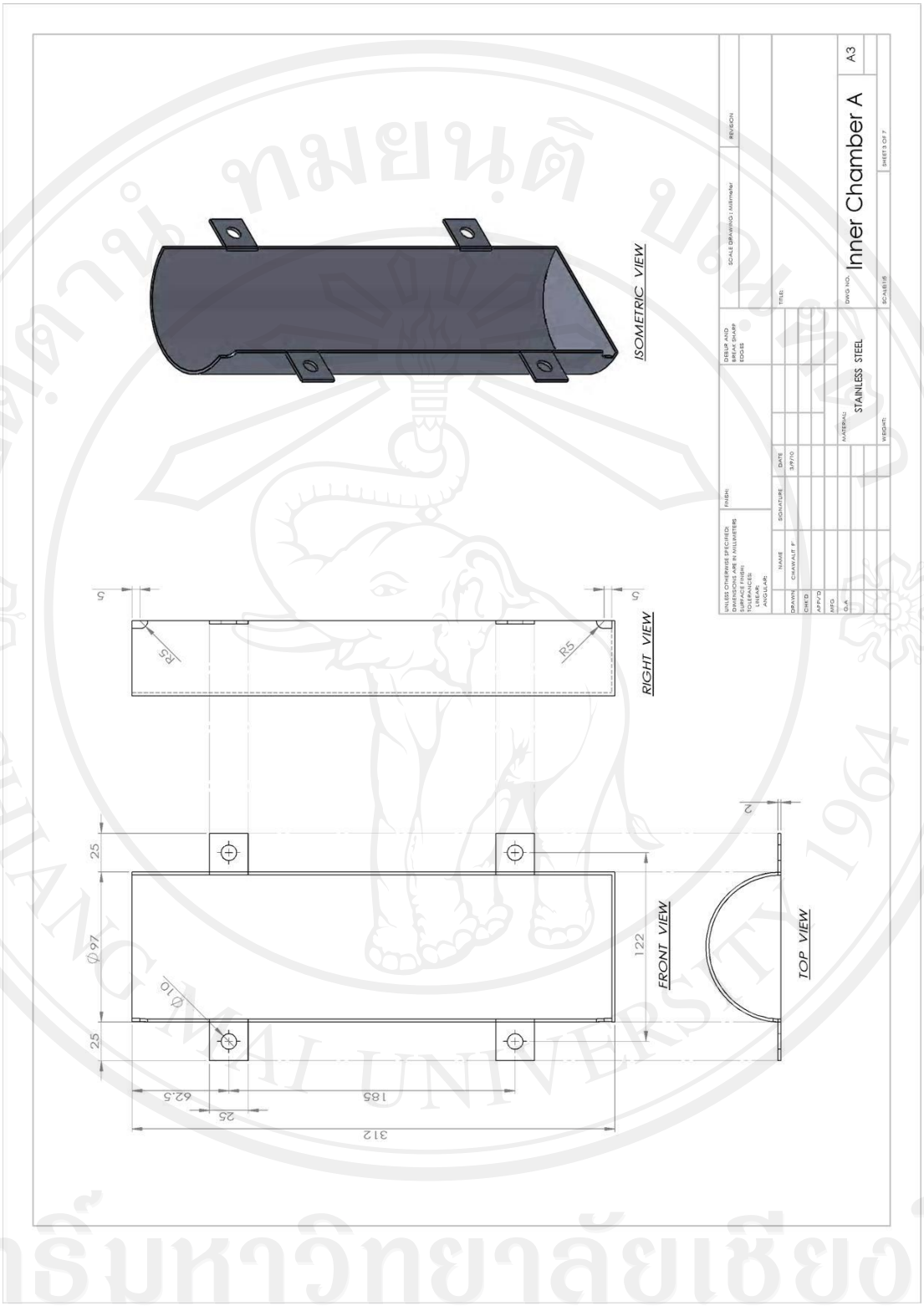

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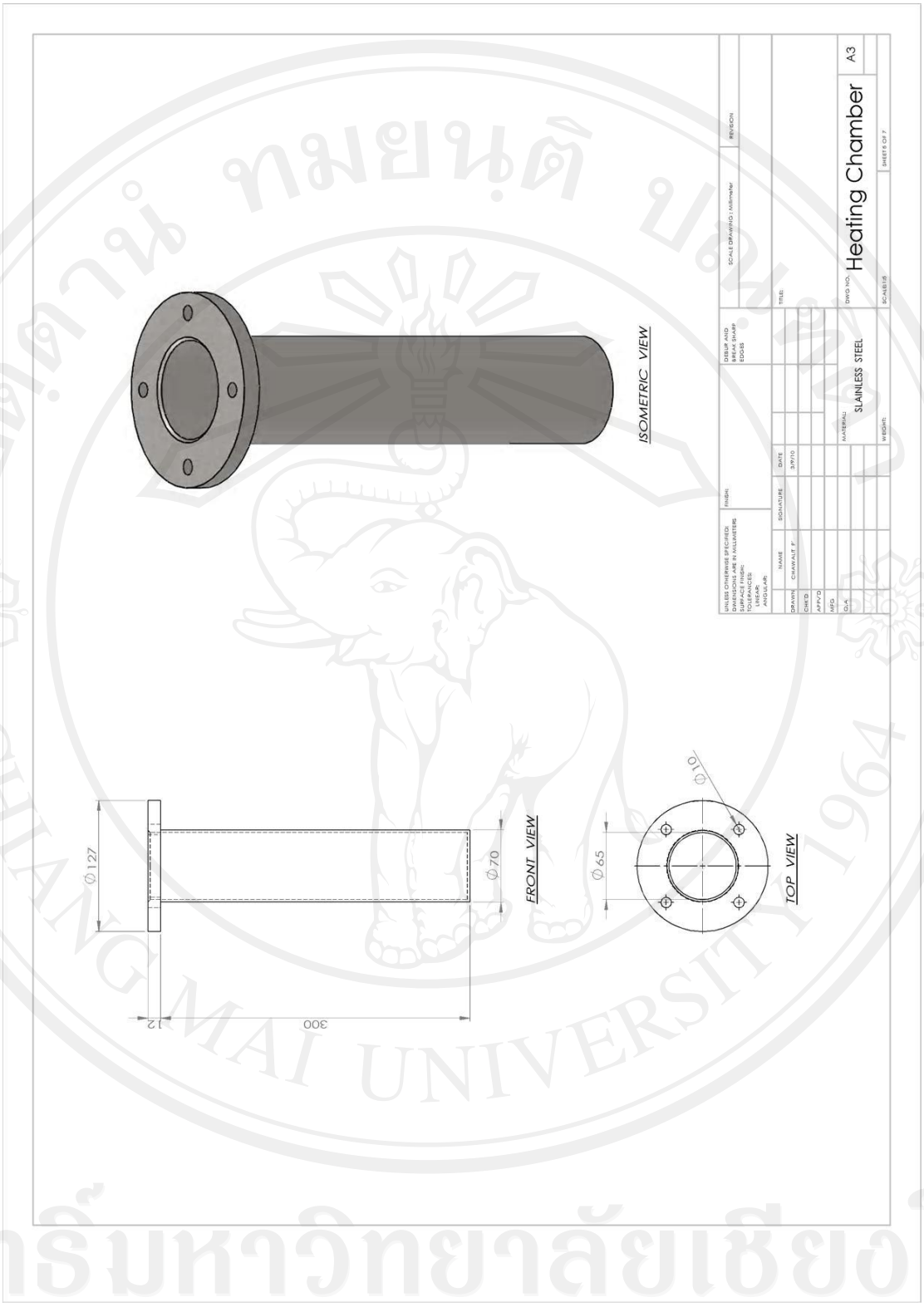

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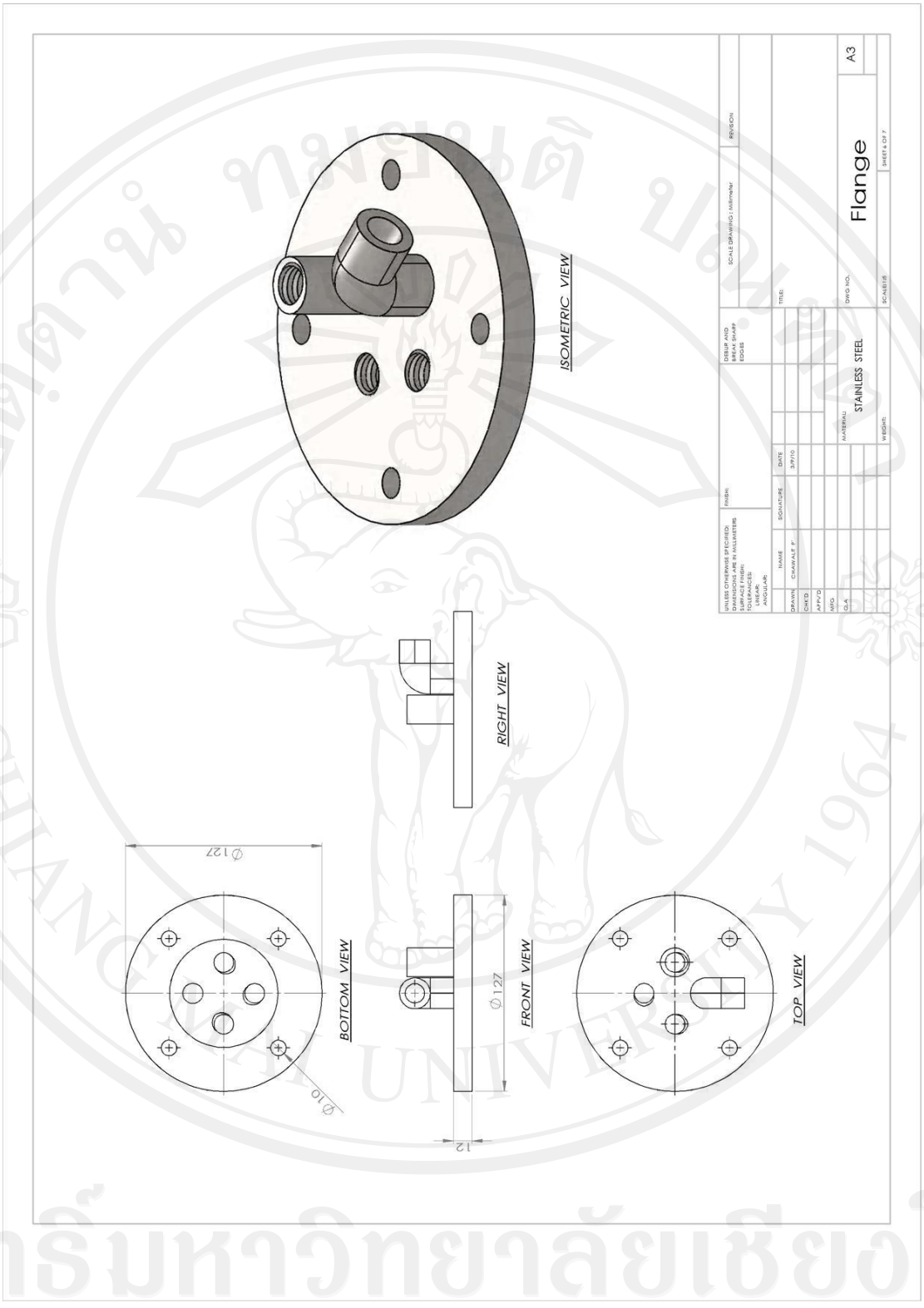

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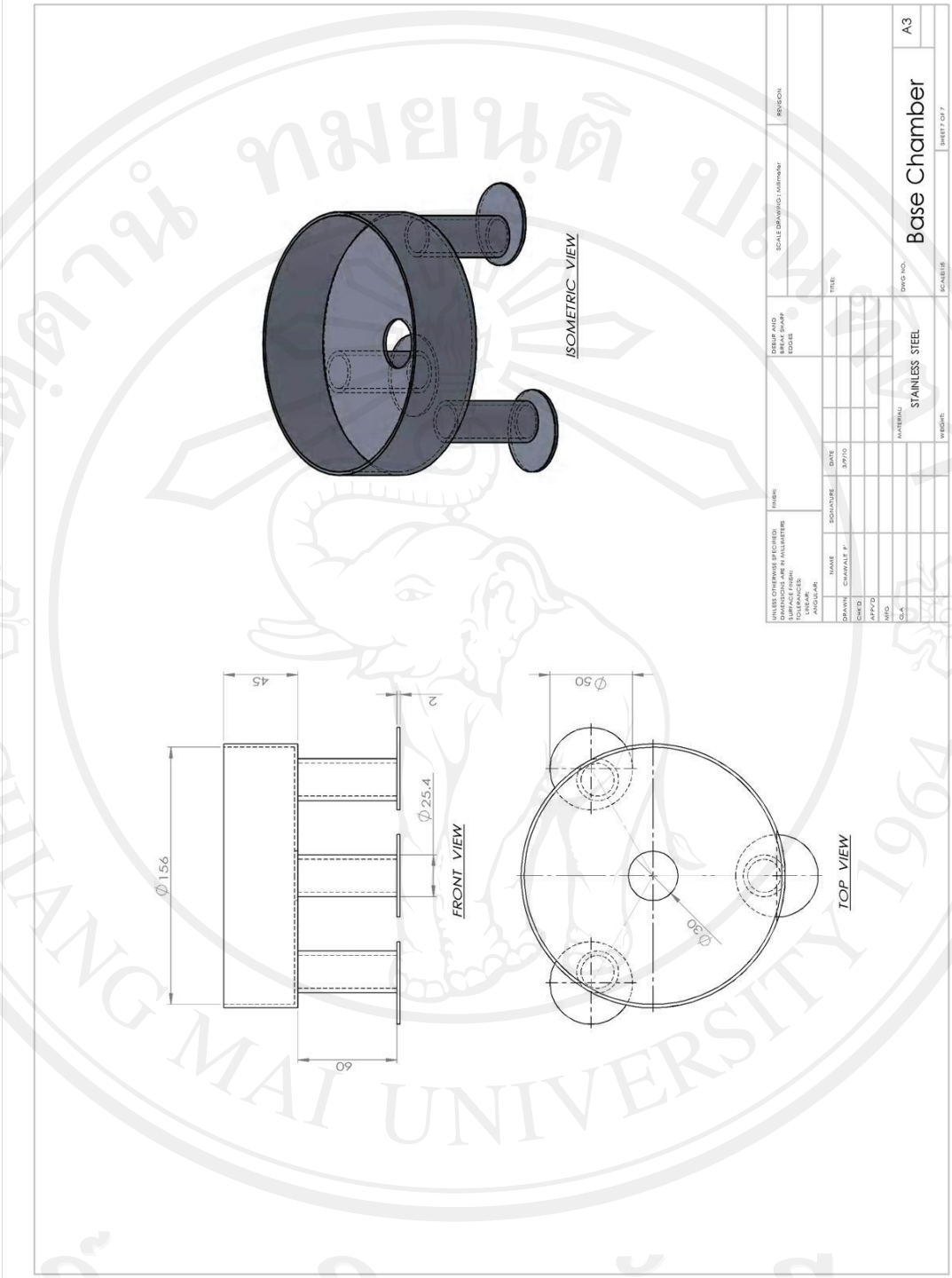

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รูป ข-1 (ก)-(ช) แสดงลักษณะการออกแบบและขนาดของเตาปฏิกรณ์ที่มีขนาด 1 ลิตร

ภาคผนวก ค

ข้อมูลการวิเคราะห์ HTC

ตาราง ค-1 ผลของการทดสอบขนาดอนุภาคที่มีผลต่อค่าความร้อนในเวลา 1 ชั่วโมง

ขนาดอนุภาค (mm)	ครั้งที่ 1 (cal/g)	ครั้งที่ 2 (cal/g)	ครั้งที่ 3 (cal/g)	เฉลี่ย (cal/g)	ค่าความร้อน (MJ/kg)
น้อยกว่า 3.0	5098.48	5253.66	5330.77	5227.64	21.89
3.0 – 5.0	4958.79	4934.74	4829.27	4907.60	20.55
มากกว่า 5.0	4750.18	5035.08	4730.35	4838.54	20.26

ตาราง ค-2 ผลของปัจจัยด้านขนาดของอนุภาคเทียบเวลาการเกิดปฏิกิริยาในเตาปฏิกรณ์

ขนาดอนุภาค (mm)	เวลา (h)	ครั้งที่ 1 (cal/g)	ครั้งที่ 2 (cal/g)	ครั้งที่ 3 (cal/g)	เฉลี่ย (cal/g)	ค่าความร้อน (MJ/kg)
น้อยกว่า 3.0	1	5098.48	5253.66	5330.77	5227.64	21.89
	2	5542.05	5710.77	5571.74	5608.10	23.48
	4	6605.71	6468.19	6543.77	6539.22	27.38
	6	6881.30	6810.77	-	6846.03	28.66
3.0 – 5.0	1	4958.79	4934.74	4829.27	4907.60	20.55
	2	5479.16	5486.06	5421.78	5462.33	22.87
	4	5908.22	6210.64	6225.69	6114.85	25.60
	6	6864.21	6790.86	-	6827.54	28.59
มากกว่า 5.0	1	4750.18	5035.08	4730.35	4838.54	20.26
	2	5390.17	5361.24	5445.74	5399.04	22.60
	4	5867.01	5754.06	5879.81	5822.62	24.42
	6	6999.47	6846.48	-	6922.98	28.99

ตาราง ค-3 ผลของตัวเร่งปฏิกิริยาในอัตราส่วน 1 ต่อ 1 เทียบกับเวลาการเกิดปฏิกิริยาในเตาปฏิกรณ์

ขนาดอนุภาค (mm)	เวลา (h)	ครั้งที่ 1 (cal/g)	ครั้งที่ 2 (cal/g)	ครั้งที่ 3 (cal/g)	เฉลี่ย (cal/g)	ค่าความร้อน (MJ/kg)
น้อยกว่า 3.0	1	5777.90	5790.04	5876.01	5814.65	24.34
	2	6700.62	6442.82	6656.68	6600.04	27.63
	4	6720.08	6785.89	6556.46	6687.48	28.00
	6	6871.75	6951.72	-	6911.74	28.94
3.0 – 5.0	1	5850.68	5897.61	5889.45	5879.24	24.62
	2	6819.81	6680.92	6615.39	6705.37	28.07
	4	7289.84	7230.84	7296.02	7272.23	30.45
	6	7198.89	7238.92	-	7218.91	30.22
มากกว่า 5.0	1	5740.90	5986.83	5986.25	5904.66	24.72
	2	6742.05	6848.80	6661.88	6750.91	28.26
	4	7212.67	7257.02	7418.91	7296.20	30.55
	6	7264.41	7314.38	-	7289.40	30.52

ตาราง ค-4 แสดงร้อยละผลได้ ของถ่านชีวภาพ ณ สภาวะต่างๆ ภายใต้อุณหภูมิและความดันเดียวกันของกระบวนการทดลอง

ขนาดอนุภาค (mm)	เวลา (h)	อัตราส่วนชีวมวลต่อตัวเร่งปฏิกิริยา	น้ำหนักก่อนการทดลอง (g)	น้ำหนัก หลังการทดลอง (g)	ผลได้ (yield)
น้อยกว่า 3.0	1	1/0	19.92	11.42	0.57
		1/1	20.00	10.54	0.53
		1/2	20.06	10.52	0.51
	2	1/0	11.34	9.14	0.55
		1/1	20.20	10.77	0.47
		1/2	19.98	10.03	0.53
		1/0	20.20	11.10	0.50

	4	1/1	20.40	10.42	0.51
		1/2	20.20	10.42	0.55
	6	1/0	20.04	11.80	0.59
		1/1	20.08	10.54	0.48
		1/2	20.02	10.06	0.52
3.0-5.0	1	1/0	20.06	11.74	0.59
		1/1	20.08	10.44	0.52
		1/2	19.97	10.21	0.59
	2	1/0	20.22	11.28	0.56
		1/1	20.04	10.44	0.52
		1/2	20.07	10.10	0.57
	4	1/0	20.26	10.98	0.54
		1/1	20.06	10.09	0.50
		1/2	20.20	10.86	0.55
	6	1/0	20.04	11.49	0.57
		1/1	20.02	10.32	0.52
		1/2	20.00	10.08	0.52
มากกว่า 5.0	1	1/0	20.02	11.42	0.57
		1/1	20.08	10.44	0.52
		1/2	20.23	10.14	0.54
	2	1/0	20.06	11.04	0.55
		1/1	20.04	10.44	0.52
		1/2	20.20	10.19	0.56
	4	1/0	20.42	10.16	0.50
		1/1	20.06	10.09	0.50
		1/2	19.98	10.08	0.54
	6	1/0	20.00	11.72	0.59
		1/1	20.02	10.32	0.52
		1/2	20.00	10.29	0.52

ตาราง ค-5 การสูญเสียความร้อนของพื้นผิวผนังที่ไม่ได้หุ้มฉนวน (kW/m)

อุณหภูมิผิวผนัง (oC)	70	80	100	120	140	160	180	200	220	240	260	280	300	320
ความสูงผนัง (m)	**การสูญเสียความร้อนของพื้นผิวผนังที่ไม่ได้หุ้มฉนวน (kW/m)**													
0.50	0.17	0.24	0.37	0.53	0.71	0.92	1.15	1.40	1.68	2.00	2.35	2.74	3.16	3.62
0.60	0.21	0.29	0.46	0.65	0.87	1.11	1.39	1.70	2.04	2.43	2.85	3.31	3.83	4.39
0.70	0.25	0.34	0.54	0.77	1.02	1.31	1.64	2.00	2.41	2.86	3.35	3.90	4.50	5.16
0.80	0.29	0.39	0.62	0.88	1.18	1.52	1.89	2.31	2.78	3.29	3.86	4.49	5.18	5.94
0.90	0.33	0.44	0.71	1.00	1.34	1.72	2.15	2.62	3.15	3.73	4.38	5.09	5.87	6.72
1.00	0.37	0.50	0.79	1.12	1.50	1.93	2.40	2.93	3.52	4.18	4.90	5.69	6.56	7.51
1.10	0.41	0.55	0.88	1.25	1.67	2.14	2.66	3.25	3.90	4.62	5.42	6.29	7.26	8.31
1.20	0.45	0.61	0.96	1.37	1.83	2.35	2.92	3.57	4.28	5.07	5.94	6.90	7.96	9.11
1.30	0.49	0.66	1.05	1.49	2.00	2.56	3.19	3.89	4.67	5.53	6.47	7.52	8.66	9.91
1.40	0.53	0.72	1.14	1.62	2.16	2.77	3.45	4.21	5.05	5.98	7.01	8.13	9.37	10.72
1.50	0.57	0.77	1.23	1.75	2.33	2.99	3.72	4.54	5.44	6.44	7.54	8.75	10.08	11.54
1.60	0.61	0.83	1.32	1.87	2.50	3.20	3.99	4.86	5.83	6.90	8.08	9.38	10.80	12.35
1.70	0.65	0.89	1.41	2.00	2.67	3.42	4.26	5.19	6.22	7.37	8.62	10.00	11.52	13.17
1.80	0.70	0.94	1.50	2.13	2.84	3.64	4.53	5.52	6.62	7.83	9.17	10.63	12.24	14.00
1.90	0.74	1.00	1.59	2.26	3.01	3.86	4.80	5.85	7.02	8.30	9.71	11.26	12.97	14.83
2.00	0.78	1.06	1.68	2.39	3.19	4.08	5.08	6.19	7.41	8.77	10.26	11.90	13.69	15.66
2.10	0.82	1.12	1.77	2.52	3.36	4.30	5.35	6.52	7.81	9.24	10.81	12.54	14.43	16.49
2.20	0.87	1.18	1.86	2.65	3.53	4.53	5.63	6.86	8.22	9.72	11.37	13.18	15.16	17.33
2.30	0.91	1.23	1.96	2.78	3.71	4.75	5.91	7.20	8.62	10.19	11.92	13.82	15.90	18.17

ตาราง ค-6 การสูญเสียความร้อนของพื้นผิวท่อหลังหุ้มฉนวนใยแก้ว (W/m)

เส้นผ่านศูนย์กลางท่อ		อุณหภูมิก่อนหุ้ม (°C)	70	80	90	100	120	140	160
De	Di	อุณหภูมิหลังหุ้มเฉลี่ย (°C)	40.19	41.34	42.43	42.51	44.28	43.55	44.23
mm.	mm.	Pipe size (in.)	การสูญเสียความร้อนหลังหุ้มฉนวนที่ความหนาที่เหมาะสม (W/m)						
10.29	6.83	1/8"	4.27	5.60	6.96	8.35	11.16	12.20	14.63
13.72	9.25	1/4"	4.83	6.35	7.91	8.46	11.25	13.73	16.48
17.75	12.52	3/8"	5.44	7.17	8.94	9.51	12.67	15.41	18.51
21.34	15.8	1/2"	5.97	7.87	9.83	10.40	13.86	16.82	20.22
26.67	20.93	3/4"	6.72	8.88	11.09	11.67	15.56	18.84	22.65
33.4	26.64	1"	7.64	10.10	12.64	13.20	17.63	21.28	25.61
42.16	35.05	1.1/4"	8.80	11.65	14.60	15.11	20.21	24.35	29.32
48.26	40.89	1.1/2"	9.59	12.72	15.94	13.85	18.38	26.44	31.85
60.33	52.5	2"	11.13	14.78	18.55	15.81	21.00	30.50	36.76
73.03	62.71	2 1/2"	10.54	13.84	17.22	17.81	23.68	29.61	35.59
88.90	77.93	3"	12.06	15.86	19.74	20.25	26.95	33.76	40.59
101.60	90.12	3 1/2"	13.26	17.45	21.73	22.17	29.52	37.04	44.54
114.30	102.3	4"	14.46	19.04	23.72	24.06	32.05	40.29	48.46
141.30	128.2	5"	16.98	22.38	27.90	24.18	32.09	47.14	48.57
168.27	154.1	6"	19.49	25.70	32.06	27.41	36.39	53.92	55.27
219.08	202.7	8"	24.18	31.91	39.84	33.37	44.35	56.42	59.79
273.05	254.5	10"	29.15	38.49	48.07	39.57	52.63	67.38	71.06
323.85	304.80	12"	33.81	44.66	55.81	45.32	60.30	77.64	81.62

ตาราง ค-7 การสูญเสียความร้อนของท่อหลังหุ้มฉนวนใยหิน (W/m)

เส้นผ่านศูนย์กลางท่อ		อุณหภูมิก่อนหุ้ม (°C)	90	100	120	140	160	180	200
De	Di	อุณหภูมิหลังหุ้มเฉลี่ย (°C)	42.72	42.81	44.65	43.91	44.62	45.80	46.94
mm.	mm.	Pipe size (in.)	การสูญเสียความร้อนหลังหุ้มฉนวนที่ความหนาที่เหมาะสม (W/m)						
10.29	6.83	1/8"	7.24	8.69	11.62	12.74	15.29	17.84	20.41
13.72	9.25	1/4"	8.22	8.78	1168	14.34	17.22	20.11	23.01
17.75	12.52	3/8"	9.29	9.86	13.14	16.08	19.33	22.58	25.85
21.34	15.8	1/2"	10.20	10.78	14.37	17.56	21.11	24.68	28.26
26.67	20.93	3/4"	11.51	12.08	16.13	19.66	23.65	27.66	31.69
33.4	26.64	1"	13.11	13.65	18.25	22.20	26.72	31.28	35.85
42.16	35.05	1.1/4"	15.13	15.62	20.91	25.40	30.60	35.83	41.08
48.26	40.89	1.1/2"	16.52	14.37	19.09	27.57	33.23	38.92	44.64
60.33	52.5	2"	19.22	16.39	21.79	31.79	38.34	44.94	51.56
73.03	62.71	2 1/2"	17.90	18.46	24.56	30.91	37.16	43.44	49.74
88.90	77.93	3"	20.52	20.98	27.94	35.23	42.37	49.55	56.76
101.60	90.12	3 1/2"	22.59	22.96	30.59	38.64	46.49	54.38	62.31
114.30	102.3	4"	24.64	24.91	33.21	42.03	50.58	59.18	67.82
141.30	128.2	5"	28.98	25.08	33.30	49.17	50.73	59.25	67.81
168.27	154.1	6"	20.20	26.66	33.29	28.42	37.76	56.24	57.73
219.08	202.7	8"	25.06	33.10	41.36	34.58	45.98	58.90	62.47
273.05	254.5	10"	30.19	39.91	49.90	40.99	54.54	70.32	71.25
323.85	304.80	12"	35.01	46.31	57.92	46.92	62.47	81.03	85.27

ห้องปฏิบัติการกลาง อาคารเฉลิมพระเกียรติชั้น 2 คณะเกษตรศาสตร์ มหาวิทยาลัยเชียงใหม่
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No. 5311/85

ใบรายงานผลการวิเคราะห์

วันรับตัวอย่าง : 3 พฤศจิกายน 2553 **วันรายงานผล** : 11 พฤศจิกายน 2553

ชนิดตัวอย่าง : ตัวอย่างน้ำ

ชื่อผู้ส่งตรวจ : นายกวินทร์ ศุภวิทยโยธิน **ที่อยู่** : คณะวิศวกรรมศาสตร์ (วิศวกรรมพลังงาน)

ผลวิเคราะห์น้ำ

ลำดับที่	รายการวิเคราะห์	หน่วย	Lab No.5311009 น้ำส้มควันไม้
1	pH	-	3.41
2	Electrical Conductivity(EC)	mS/cm	1.83
3	Total Nitrogen (N)	%	0.01
4	Phosphorus (P)	mg/L	51.69
5	Potassium (K)	mg/L	1,641.89
6	Calcium (Ca)	mg/L	222.86
7	Magnesium (Mg)	mg/L	40.72
8	Iron (Fe)	mg/L	15.03
9	Manganese (Mn)	mg/L	0.75
10	Copper (Cu)	mg/L	0.00
11	Zinc (Zn)	mg/L	6.22
12	Sulfate (SO_4^-)	mg/L	11.85
13	Boron (B)	mg/L	0.39

ผลการวิเคราะห์นี้รับรองเฉพาะตัวอย่างที่ส่งตรวจเท่านั้น ห้ามนำผลการวิเคราะห์ประกอบการโฆษณาใดๆ

.....
(นางสาววรรณภัสสรณ์ มณีเป็ญ)
ผู้วิเคราะห์

.....
(ดร. ชูชาติ สันธทรัพย์)
ผู้ตรวจสอบ

หมายเหตุ : ตัวอย่างที่ส่งวิเคราะห์จะเก็บรักษาไว้เป็นเวลา 1 เดือน หากมีปัญหาโปรดติดต่อกลับ ภายใน 1 เดือนนับตั้งแต่วันได้รับผล

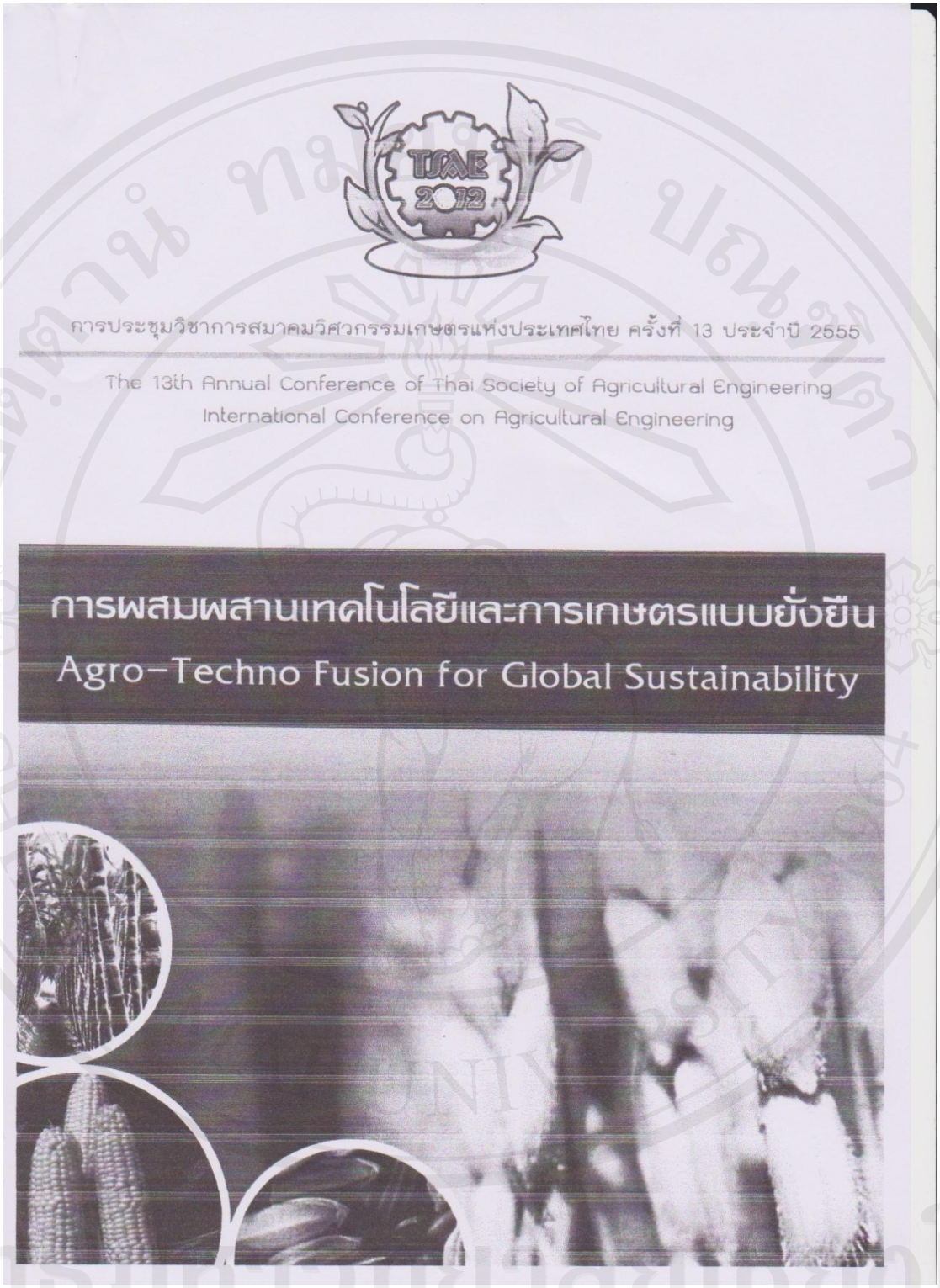
รูป ข- 2 แสดงตัวอย่างการหาค่าองค์ประกอบในสารละลาย

ภาคผนวก ง

ผลงานตีพิมพ์เผยแพร่

ชื่อผลงาน	Parametric Investigation of Biochar Production from Bamboo Using Hydrothermal Carbonization
ผู้เขียน	Kawin Supawittayayothin and Nakorn Tippayawong
ชื่องาน	GMSTEC 2010: International Conference for a Sustainable Greater Mekong Subregion
ผู้จัด	Faculty of Science King Mongkut's University of Technology Thonburi
วันที่จัดงาน	26-27 August 2010
สถานที่จัดงาน	The Imperial Queen's Park Hotel, Bangkok, Thailand

ชื่อผลงาน	Characterization of biochar from hydrothermal carbonization of bamboo
ผู้เขียน	Daniel Schneider, Marina Escala, Kawin Supawittayayothin, Nakorn Tippayawong
ชื่องาน	International Journal of Energy and Environment (IJEE) Volume 2, Issue 4, 2011, pp.647-652



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Parametric Investigation of Biochar Production from Bamboo Using Hydrothermal Carbonization

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Abstract

This paper presents a parametric investigation of biochar production from bamboo using hydrothermal carbonization. Laboratory scale, experimental setup to produce carbonaceous materials at high pressure was designed and built. The suspended biomass sample in subcritical water was subjected to hydrothermal carbonization at 220°C, 2.2 MPa, at biomass to oxalic catalyst ratio of 1:0, 1:1, and 1:2. Particle sizes of less than 3.0-10.0 mm was incubated in a closed vessel for 1 to 6 h. The end products were in solid and liquid phases, Analyses of both phrases were carried out using bomb calorimetry, scanning electron microscopy and atomic absorption spectroscopy. The experimental results revealed that oxalic acid increased the higher heating value (HHV) of the biochar while different sizes of particle did not affect the HHV. The coal-like biochar was found to have rough surface and porous structure. The aqueous solution was found to contain a high concentration of nutrients, especially nitrogen, phosphorus, and potassium.

Keywords: Biomass, Char, Hydrothermal carbonization, Subcritical water, Renewable energy.

1. Introduction

Hydrothermal carbonization (HTC) is a coalification-imitating process which tends to produce coal from high moisture-containing waste materials. Since HTC is a water-based process, wet biomass, and organic wastes can be used as starting materials without being dried. To progress HTC, specific condition adjusted from the condition of biofuel production (300 °C, 10 MPa) has to be achieved because other chemical reactions yielding undesirable products can take place under milder or harsher conditions. For example, water-soluble substances in starting materials can be extracted at 100 °C. Hydrolysis of cellulose, hemi-cellulose, protein, and many other biopolymers can proceed at temperature higher than 150 °C. At 200 °C and 1 MPa, solid biomass becomes slurry. In contrast, biomass gas can be acquired at a critical point using catalyst [1,2].

HTC is a promising process that provides several advantages. One of them is that undesirable byproducts particularly CO_2 can be avoided. This is a big difference from most of the other biomass energy processes. There are also

very promising ways to use HTC to generate energy source. [3]

Char properties including porosity, and electrical conductivity have been studied for industrial purposes. Liu et at. showed that the porosity of hydrothermal carbonized char can be increased by physical adsorption [4,5,6]. An increase of electrical conductivity of rice husk was found to be approachable using different concentrations of H_2SO_4 at 95 °C [7]. Moreover, char obtained from algae treated at 200 °C (<2 mPa) for less than 1 hour was able to supplement soil nutrients [6,8,9].

In this study, the ratio of biomass per oxalic catalyst, biomass size, and reaction time were varied to discover factors affecting heat value of chars produced from bamboo HTC and properties of byproducts solution.

2. Experimental

2.1 Raw materials

Bamboo (Dendrocalamus asper) was collected from a local bamboo furniture factory. The raw material was cut, milled and sieved to particle sizes between 3.0-10.0 mm, used as the feedstock for experimental runs. It was subsequently air dried to low moisture content (< 10 %). All of the materials were stored in plastic bags at room temperature until being used in the experimentation. Its properties obtained from chemical analysis in a previous study are shown in Table 1.

2.2 HTC reactor

Experimental runs on HTC of the bamboo were performed in a batch reactor, as shown in Fig 1. The HTC reactor was 0.6 m high and inside diameter of 50 mm. It was made from stainless steel cylinder, surrounded by a 3-kW electric heater coil, thick insulating wool, and a 3 mm thick steel sheet. It had a 30 mm thick steel cover, equipped with temperature and pressure sensors, as well as pressure relief valve.

2.3. Hydrothermal treatment

HTC of bamboo was carried out according to the following procedures. Particle size between 3.0-10.0 mm of bamboo, at biomass to catalyst ratio of 1:0, 1:1, and 1:2 were used. Prior to the test, pre-weighed batches of bamboo materials (20 g) were dispersed in distilled water (300 mL) and stirred for 4 h. The mixture was then loaded into the HTC reactor. The cover was put in place, checking that the reactor was air tight. The closed reactor was then heated to 220°C, 2.2 MPa for 1 to 6 h, before cooling down to room temperature.

Table 1. Chemical analysis of bamboo

Property	Unit	Value
Proximate analysis (% w/w dry basis)		
Moisture	[%]	5.7
Volatile	[%]	74.7
Fixed carbon	[%]	14.1
Ash	[%]	5.5
Ultimate analysis (% w/w dry basis)		
Carbon	[%]	45.7
Hydrogen	[%]	4.3
Oxygen	[%]	49.7
Nitrogen	[%]	0.3
Higher heating value	[MJ/kg]	16.8

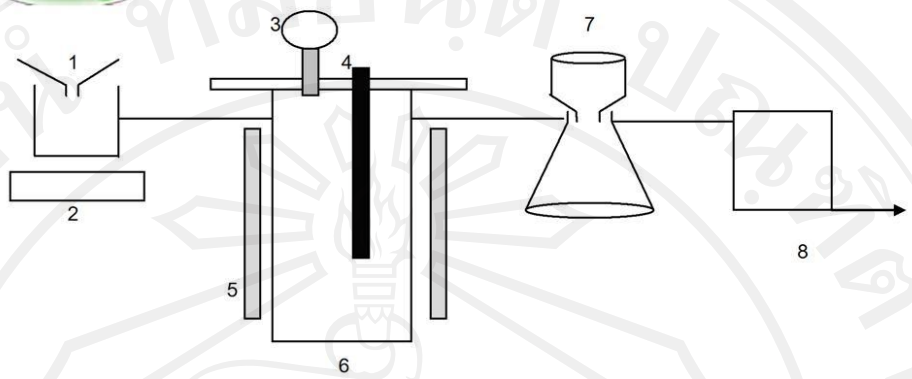


Figure 1. Schematic diagram of the HTC reactor: (1) mixture, (2) hotplate stirrer, (3) pressure sensors, (4) thermocouple, (5) electric heater coil, (6) reactor, (7) vacuum filtration and (8) oven

Reaction temperatures were measured by a thermocouple inserted through its cover. The temperature and pressure were recorded manually at a regular interval of 10 min. At the end of each experiment, the solid chars and process water were collected and weighed to determine the mass balance. The chars were filtered, followed by drying in an oven at 105°C for 4 h.

2.4 Characterization methods

Surface morphology was studied by scanning electron microscopy. Char imaging was carried out using a JEOL JSM-6335F Field Emission Scanning Electron Microscope. In SEM analysis, char samples were selected and imaged randomly to minimize bias. Magnifications between 1000X and 3,000X were typically used. Additionally, the heating value of the dried bamboo, and the resulting char were determined using a Parr bomb calorimeter. It was reported as gross heat of combustion at constant volume.

Atomic absorption analysis was used to determine the nutrient content of the processed water. The target elements in this study were N, P, and K. Exactly 25 mL of each sample were used for analysis. Each sample was directly heated in crucible until all carbonaceous matter was removed. The ash was then allowed to cool down, and later transferred to a flask. The ash was treated with 10 mL of concentrated HNO_3 and 30 mL of concentrated HCl. All the reagents used were of analytical grade. The mixture was digested at 60°C for at least 2-3 h or until the mixture appeared to have no residue. Digested samples were filtered and their volume were adjusted to 100 mL by adding solution of 1.0% HNO_3 in deionized double distilled water. High purity of standard stock solutions of 1000 ppm (mg/L) were prepared for different metals. The atomic absorption determinations were measured with a Perkin-Elmer Corporation AA analyst 100 instrument. A data were sampled and processed automatically via a personal computer using AA Win/Lab software. The spectrometer was operated in the absorption mode (absorbance readings), using standard solutions for calibration. For each element determination, the recommended wavelengths were set. Burner

position as well as flame conditions (C_2H_2, air, N_2O) were optimized using a standard solution. Deionized water was atomized after each reading of standard or sample. The calibration curves were prepared by plotting the absorbance versus concentration for each standard. From calibration graphs, concentrations of a sample were calculated by comparing the absorbance of the sample solution with that of the standard solution. All analytical determinations were performed in triplicate and average results were presented.

3. Results and Discussion

3.1 Temperature and pressure evolution

A temperature measurement was possible using a thermocouple inserted directly into the closed reaction chamber. Temperature and the corresponding pressure profiles are illustrated in Fig. 2, which shows that the heating arrangement was able to provide the reaction temperature rapidly (less than 50 min), and maintain at the controlled HTC temperature and pressure within a small degree of fluctuation throughout the test runs.

3.2 Effect of particle size

To investigate parameters affecting heating value of biochar production, between less than 3.0-10.0 mm particle sizes of bamboo were processed in a 1 liter closed reactor for a time variation between one to six hours. Results are show in Fig. 3.

The findings demonstrate a direct proportional trend between reaction time and heating value. With six hours of incubation, chars were found to exhibit maximum heat value of 28.8 MJ/kg for less than 3 mm diameter, 28.7 MJ/kg for 3-5 mm diameter, and 29.3 MJ/kg for more than 5 mm diameter. However, the production potential calculable from energy equilibrium will appear to be dropped at a certain point possibly due to the change of product's structure causing atomic ratio (O/C) change [5].

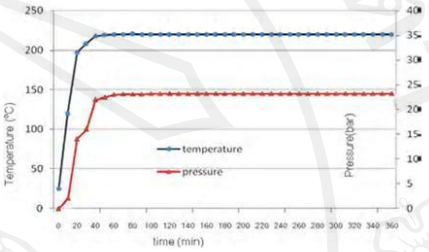


Figure 2. Evolution of reaction temperature and pressure inside the HTC reactor

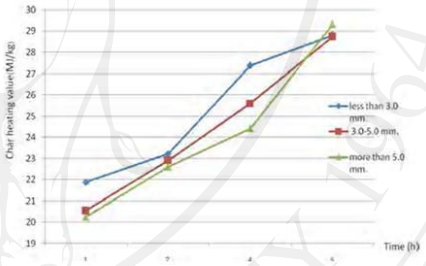


Figure 3. Effect of particle sizes on char HHV

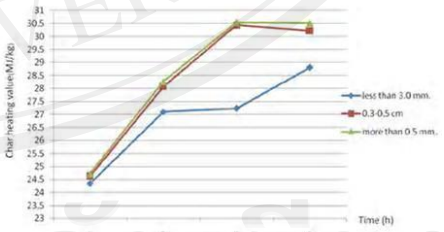
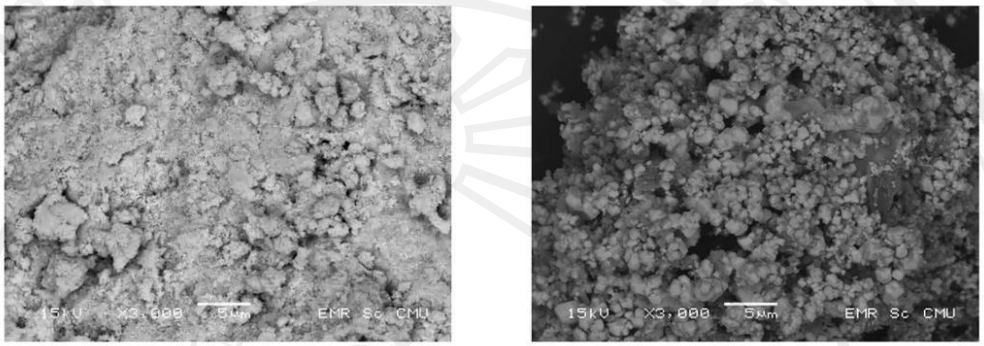


Figure 4. Effect of biomass to catalyst ratio on biochar HHV



(a) (b)

Figure 5. SEM images of (a) raw bamboo and (b) 4 h, (B/C) 1:0 biochar from HTC of bamboo

3.3 Effect of catalyst

To study the catalytic effect of oxalic acid on the bamboo HTC, less than 3 mm diameter of bamboo particle was used in the experimental process. Results are show in Fig. 4. The data reveal that an addition of catalyst under a variation of reaction time rapidly increases heat value of the char product until 4 hours of incubation.

Hydrothermal treatment of biomass leads to the formation of solid char particles, potentially a coal-like fuel. In this work, about 45% of original raw bamboo mass was recovered as HTC char. Images of raw bamboo and HTC char are shown in Fig. 5. The surface property is important for char's activity. From the SEM results, it was observed that the HTC char had rougher surface than the raw bamboo. Porous structures were formed on the surface. The HTC char was observed to contain more porous structure than the original bamboo samples.

3.4 Nutrients in process water

It is generally known that water plays significant roles as a solvent and a reactant in the HTC process. The liquid phase is expected to contain a high load of organics and inorganics. Atomic absorption analysis of the process water from bamboo carbonization was carried out. Results are shown in in Table 2. A drop in pH of the process water was observed after HTC reaction. It is noted here that original pH of deionized water was at neutral. The liquid phase was found to be acidic which can be explained by the formation of a variety of organic acids that typically occur during the HTC process

The nutrient content of the processed water was analyzed in order to inspect its value as a useful fertilizer. The liquid phase was found to contain high values of potassium and nitrogen, while the phosphorus was relatively low [8].

Table 2. Elemental analysis of bamboo char under different conditions

Particle size (mm)	> 5.0	5.0 - 3.0	< 3.0	< 3.0	<3.0
Biomass to catalyst ratio (B/C)	1/0	1/0	1/0	1/1	1/2
Carbonization					
Temperature (°C)	220	220	220	220	220
Time (h)	4	4	4	4	4
Char					
heating value (MJ/kg)	24.4	25.6	27.4	27.7	27.8
Process water					
Total nitrogen (mg/L)	110	100	100	105	105
Phosphorus (mg/L)	50	55	52	58	54
Potassium (mg/L)	1720	1580	1642	1816	1838
pH	3.2	3.5	3.5	3.0	2.8

4. Conclusion

In this study, carbonaceous samples have been prepared by hydrothermal carbonization of bamboo. Preliminary study has shown that a thorough carbonization process can take place during hydrothermal treatment at relatively mild conditions, evidenced by the stark change in the heating value of the samples, which is a consequence of the rearrangement of the chemical bounds, especially those between carbon, oxygen and hydrogen. Oxalic acid is a good catalyst to increase HHV of the biochar. However, the variation of particle sizes between 3-10 mm does not significantly affect the HHV at 6 h. The HTC treatment can develop rough surface and porous structure of the obtained biochar. The coal-like solid particles produced during the treatment may be used as fuels, or alternatively suitable for adsorption purposes because of its structure. Additionally, limited analysis of the process water suggested that there may be a vast amounts of compounds dissolved in the liquid phase. The liquid phase contained nutrients that may be useful as fertilizer.

5. Acknowledgement

Supports from the Thailand Research Fund and the Department of Mechanical Engineering, Chiang Mai University are gratefully acknowledged.

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Characterization of biochar from hydrothermal carbonization of bamboo

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Abstract
This paper presents a preliminary investigation on producing biochar from bamboo using a technique of hydrothermal carbonization. Laboratory scale experimentation to produce carbonaceous materials was carried out. The suspended biomass samples in water were subjected to hydrothermal carbonization at 220°C, 2.2 MPa in a closed vessel for six hours. The resulting products were in solid and liquid phase. The coal-like biochar was found to have rough surface and porous structure. The aqueous solution was found to contain a high concentration of nutrients, especially nitrogen, phosphorus, and potassium. The study shows that bamboo is an interesting and adequate biomass for the production of biochar with several applications including carbon sequestration.

Keywords: Biomass; Char; Hydrothermal carbonization; Renewable energy.

1. Introduction

With the global warming in the centre of international concern and discussions, a profound review in energy policy is being conducted. In this arena, ideas for alternatives to ever-decreasing reserves of fossil fuels as well as measures to decelerate or reduce the CO_2 emissions are urgently required. The efficient management of biomass, for instance to produce biofuels, is one of the most interesting aspects under investigation in order to achieve an environmentally-clean and CO_2-neutral solution.

A possible method to convert biomass into a biofuel is hydrothermal carbonization, also known as HTC. The first experiments involving the HTC process were already performed during the first half of the twentieth century and were aimed at understanding the mechanism of natural coalification [1, 2] although it was not until recently that this mechanism was proposed for the production of biofuels [3]. The HTC process is relatively simple, requiring mainly a closed vessel that contains the wet biomass and that is heated to temperatures between 170 and 250°C over a period ranging from a few hours to a day [4]. The hydrothermal carbonization process includes several reaction mechanisms, such as hydrolysis, dehydration, decarboxylation, polymerization and aromatization, although the detailed reactions have been only well characterized for a few types of biomass, such as cellulose [5]. The process takes place effectively only in water and is exothermic. The products of the HTC are a solid phase or "HTC-coal" and a liquid phase, referred as process water. A small amount of gas is also produced.

In the field of biofuel research, most attention has been paid to the liquid and gaseous products, while the solid phase or char did not receive much consideration, partially explained by the fact that its energy

density is lower than for most of the liquid and gaseous energy carriers However, other characteristics, such as a relatively simple production process, also applicable in remote or low-industrialized areas, and its amenability for storage, thus allowing an efficient management of energy demand, make char an interesting focus in the biofuel development. Apart from its use as an energy carrier, HTC-coal has also potential applications as a soil conditioner, in mechanisms of CO_2 sequestration and storage, as a part of an integrative solution for biomass management and in the production of synthetic carbon-based materials with a variety of applications [3, 4, 6, 7].

The raw material used for the hydrothermal carbonization is preferably plant biomass with high content of lignin, cellulose and hemicellulose, e.g. agricultural waste or wood material [4] although a broad range of materials have been tested and effectively carbonized, such as microalgae, wastewater sludge or whey [4, 8]. One of the significant advantages of the HTC is that the biomass does not need to be dried. Biomass with water content up to 96% has been effectively carbonized. One of the key questions, therefore, is to find out which level of biomass dry weight is optimal for the carbonization of biomass in order to keep a reasonable energy balance.

In this study, bamboo was selected as a representative of biomass. Bamboo is the common term applied to a broad group of woody grasses (family Poaceae, subfamily Bambusoideae) ranging from 100 mm to 40 m in height. It encompasses 1250 species within 75 genera. It is distributed mostly in the tropics, comprising natural stands of native species. Bamboo has several interesting aspects for the HTC. It is a fast-growing plant that can grow on slopes and other areas where cultivation of wood is not possible [9]. Typical water content values are 10.4% at harvest and 2.9% after drying [10], which make it a suitable material for HTC. Thailand is abundant in lignocellulosic materials which are largely untapped, such as bamboo. Bamboo has been used for handicrafts, paper-making and construction materials as well as cultivated for edible shoots. Nowadays, it is used mostly for fiber and food within Asia. It has been suggested that non-fuel applications of bamboo biomass might be actually more profitable than energy recovery [10]. Actually, the heating value of bamboo is higher than most agriculture wastes but it is lower than most woody products. However, HTC process offers the possibility of increasing the energy density of bamboo by means of a basically exothermic and relatively simple process.

The principal objective of the present work was to investigate the amenability of bamboo for hydrothermal carbonization and to obtain biochar that is a solid energy carrier with a good process energy balance. The HTC process as well as the characteristics of the obtained char and the process water were the focus of this experimental study.

2. Methodology

2.1 Raw materials

Bamboo (*Dendrocalamus asper*) was collected from a local bamboo furniture factory. The raw material was cut, milled and sieved to particle sizes below 300 µm. It was subsequently air dried to low moisture content (< 10 %). All of the materials were stored in plastic bags at room temperature until being used in the experimentation. Its properties obtained from chemical analysis in a previous study [11] are shown in Table 1.

Table 1. Chemical analysis of bamboo

Property	Unit	Bamboo
Moisture	[%]	5.7
Volatile	[%]	74.7
Fixed carbon	[%]	14.1
Ash	[%]	5.5
Carbon	[%]	45.7
Hydrogen	[%]	4.3
Oxygen	[%]	49.7
Nitrogen	[%]	0.3
Higher heating value	[MJ/kg]	16.8

2.2 HTC reactor and test procedure

Experimental runs on HTC of the bamboo were performed in a batch reactor, as shown in Figure 1. The HTC reactor was 0.6 m high and inside diameter of 50 mm. It was made of stainless steel cylinder, surrounded by a 2-kW electric heater coil, thick insulating wool, and a 3 mm thick steel sheet. It had a 30

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mm thick steel cover, equipped with temperature and pressure sensors, as well as pressure relieve valve. Prior to the test, pre-weighed batches of bamboo materials (10 g) were dispersed in distilled water (300 mL) and stirred for 4 h. The mixture was then loaded into the autoclave. The cover was put in place, checking that the reactor was air tight. The closed reactor was then heated to 220°C, 2.2 MPa. The reaction time of six hours after reaching 220°C was used, before cooling down to room temperature. Reaction temperatures were measured by a thermocouple inserted through its cover. The temperature and pressure were recorded manually at a regular interval of 10 min. At the end of each experiment, the solid chars and process water were collected and weighed to determine the mass balance. The chars were filtered, followed by drying in an oven at 105°C for 4 h.

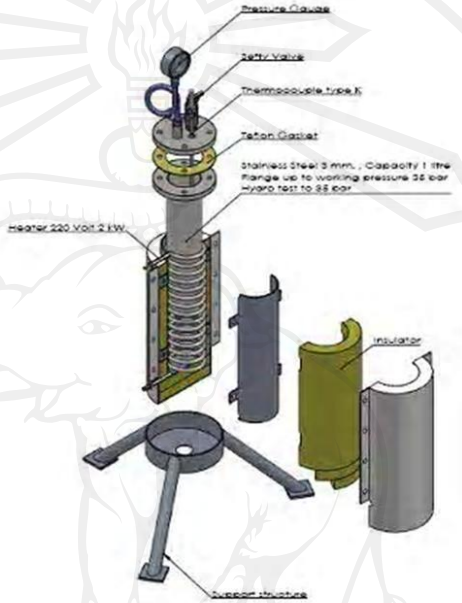

Figure 1. Schematic drawing of the HTC reactor setup

2.3 Analytical methods

The surface morphology was studied by scanning electron microscopy. Char imaging was carried out using a JEOL JSM-6335F Field Emission Scanning Electron Microscope. During the SEM analysis, char samples were selected and imaged randomly to minimize bias. Magnifications between 500X and 1,000X were typically used. Additionally, the heating value of the dried bamboo, and the resulting char were determined using a Parr bomb calorimeter. It is reported as gross heat of combustion at constant volume.

Atomic absorption analysis was used to determine the nutrient content of the process water. The target elements in this study were N, P, and K. Exactly 25 mL of each sample were used for analysis. Each sample was directly heated in crucible until all carbonaceous matter was removed. The ash was then allowed to cool down, and later transferred to a flask. The ash was treated with 10 mL of concentrated HNO_3 and 30 mL of concentrated HCl. All the reagents used were of analytical grade. The mixture was digested at 60°C for at least 2-3 hours or until the mixture appeared to have no residue. Digested samples were filtered and their volume adjusted to 100 mL by adding solution of 1.0% HNO_3 in deionized double distilled water. High purity of standard stock solutions of 1000 ppm (mg/L) were prepared for different metals. The atomic absorption determinations were measured with a Perkin-Elmer Corporation AA analyst 100 instrument and all data were sampled and processed automatically via a personal computer using AA Win/Lab software. The spectrometer was operated in the absorption mode (absorbance readings), using standard solutions for calibration. For each element determination, the recommended wavelengths were set and burner position as well as flame conditions (C_2H_2, air, N_2O) were optimized

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using a standard solution. Deionized water was atomized after each reading of standard or sample. The calibration curves were prepared by plotting the absorbance versus concentration for each standard. From calibration graphs, concentrations of a sample were calculated by comparing the absorbance of the sample solution with that of the standard solution. All analytical determinations were performed in triplicate and average results are presented.

3. Results and discussion

3.1 HTC process conditions

A temperature measurement was possible using a thermocouple inserted directly into the closed reaction chamber. Temperature and the corresponding pressure profiles are illustrated in Figure 2, which shows that the heating arrangement was able to provide the reaction temperature rapidly (less than 50 min), and maintain at the controlled HTC temperature and pressure within a small degree of fluctuation throughout the test runs.

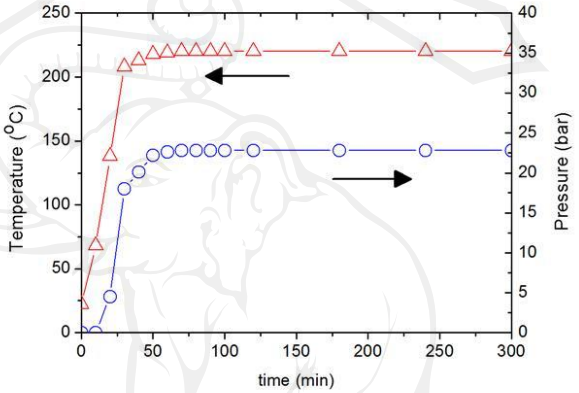

Figure 2. Evolution of reaction temperature (Δ) and pressure (○) inside the HTC reactor

3.2 Characteristics of biochar

Hydrothermal treatment of biomass leads to the formation of solid char particles, potentially a coal-like fuel. In this work, about 45% of original raw bamboo mass was recovered as HTC char. Images of raw bamboo and HTC char are shown in Figure 3. The surface property is important for char activity. From the SEM results, it was observed that the HTC char had rougher surface than the raw bamboo. Porous structures were formed on the surface. The HTC char was observed to contain more porous structure than the original bamboo samples.

Analysis of biochar energetic content showed an average heating value of 28.7 MJ/kg, which is similar to that of lignite coal. With respect to energy conversion efficiency, defined as a ratio between energy content in the biochar divided by that in the raw bamboo, it was calculated to be 76.9%. Further analysis for carbon in all HTC products should be carried out to evaluate carbon conversion efficiency of the process.

3.3 Nutrients in process water

It is generally known that water plays a significant role as a solvent and reactant in the HTC process. The liquid phase is expected to contain a high load of organics and inorganics. Atomic absorption analysis of the process water from bamboo carbonization was carried out. Results are shown in the second column in Table 2. A drop in pH of the process water was observed after HTC reaction. It is noted here that original pH of deionized water was at neutral. The liquid phase was found to be acidic which can be explained by the formation of a variety of organic acids that typically occur during the HTC process [5].

The nutrient content of the process water was analyzed in order to inspect its value as a useful fertilizer. The liquid phase was found to contain high values of potassium and nitrogen, while the phosphorus was relatively low. Also shown in Table 2, results from HTC of various biomass materials at similar conditions are compared with this work. The raw materials are cheese whey, biogas digestate, and sewage sludge.

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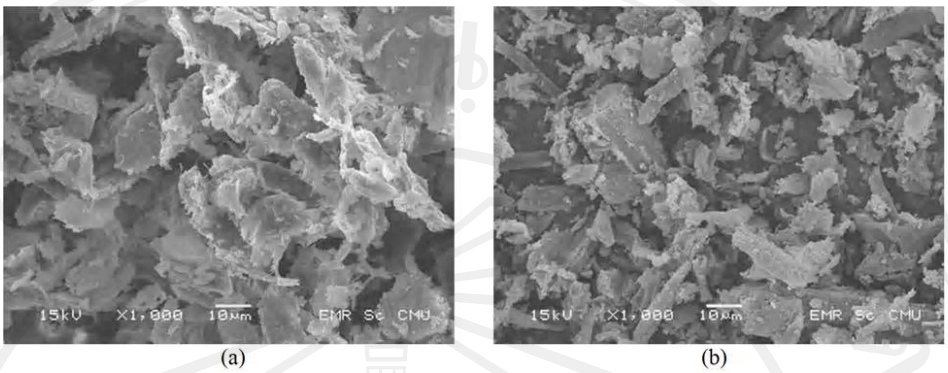

(a) (b)

Figure 3. SEM images of (a) raw bamboo and (b) biochar from HTC of bamboo

Table 2. Comparison with other biomass materials

	This work	Escala et al. [8]	Escala M. [12]	Koller C. [13]
Raw materials	Bamboo	Whey	Biogas digestate	Sewage sludge
Carbonization temp. (°C)	220	205	205	205
time (h)	6	6	6	5
Char heating value (MJ/kg)	28.7	27.1	16.4	-
Process water total nitrogen (mg/L)	100	511	2342	1230
phosphorus (mg/L)	52	226	6.4	10
potassium (mg/L)	1642	1387	6213	446
pH	3.4	3.9	9.3	5.0

4. Conclusion

In this study, carbonaceous samples have been prepared by hydrothermal carbonization of bamboo. Preliminary study has shown that a thorough carbonization process can take place during hydrothermal treatment at relatively mild conditions, evidenced by the stark change in the heating value of the samples, which is a consequence of the rearrangement of the chemical bounds, especially those between carbon, oxygen and hydrogen. The HTC treatment can develop rough surface and porous structure of the obtained biochar. The coal-like solid particles produced during the treatment may be used as fuels, or alternatively suitable for adsorption purposes because of its structure. Additionally, limited analysis of the process water suggested there may be a vast amounts of compounds dissolved in the liquid phase. The liquid phase contained nutrients that may be useful as fertilizer.

From this preliminary work, HTC appeared to be a conversion technology that is worth serious attention due to potential of its possible applications.

Acknowledgements

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