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# **Appendices**

ศูนย์วิทยทรัพยากร  
จุฬาลงกรณ์มหาวิทยาลัย

## Appendix A

### Synthesis, mechanical and Physical Properties Characterization

TableA1 Result of effect of power output and reaction time

Condition		Weight before	Weight After	%weight Increase
Time(min)	Power(watt)			
1.30	90	1.01	1.01	0
	180	1.01	1.01	0
	270	1.01	1.58	56.43
	360	1.01	1.75	73.27
	450	1.01	2.15	112.87
	540	1.01	2.05	102.97
2.10	540	1.04	1.94	86.54
2.30	90	1.01	1.1	8.91
	180	0.98	1.47	50
	270	1.01	1.83	81.19
	360	1.05	1.91	81.90
	450	1.01	1.58	56.43
	540	1.04	1.08	3.85
3.00	90	1.01	1.58	56.44
	180	1.01	1.98	96.04
	270	1.01	2.03	100.99
	360	1.01	1.97	95.05
3.30	90	1.01	2.16	113.86
	180	1.01	2.29	126.73
	270	1.01	2.41	138.61
	360	1.01	2.01	99.01

**TableA2 Result of effect of Pyridine/TsCl concentration**

Condition Eq. Pyridine/TsCl	Weight Before	Weight After	%Weight Increase
2	1.01	1.01	0
3	1.01	2.31	128.71
4	1.01	2.42	139.60
5	1.01	2.29	126.73
6	1.01	2.81	178.22

**TableA3 Result of effect of TsCl concentration and stearic acid concentration**

Eq.TsCl Eq. Stearic acid	1	2	3	4
0.5 (weight after ) (%weight increase)	1.01 0%	1.01 0%	1.39 37.62%	1.58 56.43%
1	1.01 0%	2.39 136.63%	2.16 113.86%	2.00 98.02%
2	1.34 32.67%	2.61 158.41%	3.87 283.17%	3.32 228.71%
3	1.01 0%	1.88 86.14%	2.28 125.74%	5.12 406.93%

**TableA4 Gloss value of cellulose stearate sample**

%ester	1	2	3	4	5	Sd
45.14	12.9	8.8	7.5	8.6	8.8	2.07
55.41	52.7	52.7	50.4	50.4	52.7	1.26
71.38	52.2	50.4	48.4	48.4	50.4	1.60

**TableA5 Contact angle of cellulose stearate sample**

%Ester	1	2	3	4	5	Sd
45.14	86	84	86	86	86	.89
55.41	60	62	60	60	60	.89
71.38	68	70	68	68	68	.80

**TableA6 Water absorption of cellulose stearate sample**

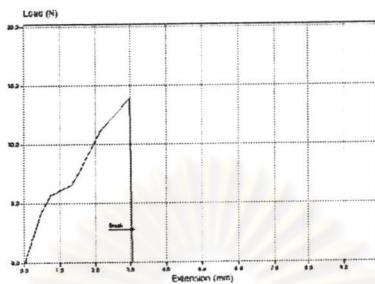
%Ester		1	2	3	sd
45.14%	Weight before	0.4339	0.4424	0.479	1.33
	Weight after	0.4957	0.5046	0.5406	
	% Wight increase	14.24291	14.22	14.26	
55.41%		0.2772	0.3886	0.3187	1.097923
		0.3619	0.5134	0.4143	
		30.55556	32.11529	29.99686	
71.38%		0.4245	0.4529	0.5528	2.517987
		0.5179	0.5474	0.6478	
		22.00236	20.86553	17.18524	

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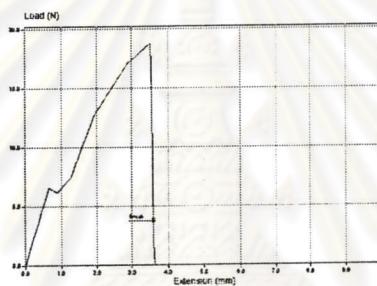
**Table A7 Mechanical properties of cellulose stearate sample**

Batch	Maximum Load (N)	Deflection at Maximum Load (mm)	Stress at Maximum Load (MPa)	% Strain at Maximum Load	Work to Max Load (J)	Stiffness (N/m)	Young's Modulus (MPa)	Load at Break (N)	Deflection at Break (mm)	Stress at Break (MPa)	% Strain at Break	Work to Break (J)
71.38%	16.81958286	4.032995125	5.606327621	4.032995125	0.042177729	9308.277824	310.2759275	3.363916572	4.083730391	1.121305524	4.083730391	0.04267169
71.38%	18.77796593	3.473868153	6.259321977	3.473868153	0.038667521	11200.2269	373.3408968	3.755593186	3.535683024	1.251864395	3.535683024	0.039474539
71.38%	16.05721504	4.124257995	5.352405014	4.124257995	0.042755884	10261.68784	342.0562612	3.211443009	4.161022586	1.070481003	4.161022586	0.043095659
71.38%	16.36581524	3.911815542	5.455271746	3.911815542	0.038989734	8926.980371	297.5660124	3.273163047	3.976150755	1.091054349	3.976150755	0.039823692
55.41%	14.35908564	3.882420826	4.786361879	3.882420826	0.035132058	9215.651487	307.1883829	2.871817128	3.926860471	0.957272376	3.926860471	0.035485577
55.41%	13.19044735	2.409485062	4.396815783	2.409485062	0.017584043	10368.18003	345.606001	2.63808947	2.461575989	0.879363157	2.461575989	0.017996305
55.41%	14.32473319	2.821590501	4.774911064	2.821590501	0.023274228	9953.542505	331.7847502	2.864946638	2.884027675	0.954982213	2.884027675	0.023970725
55.41%	13.8407155	2.910598395	4.613571832	2.910598395	0.023467361	9534.484033	317.8161344	2.768143099	2.965017019	0.922714366	2.965017019	0.023944399
45.14%	5.850811867	0.880339173	2.925405933	2.925405933	0.002927876	7177.467601	143.549352	1.170162373	0.92346053	0.585081187	2.308651326	0.003067176
45.14%	4.868811344	0.568211725	2.434405672	1.420529311	0.001465572	8251.213989	165.0242798	0.973762269	0.632659577	0.486881134	1.581648943	0.001672555
45.14%	6.157930188	1.070428753	3.078965094	2.676071883	0.003429228	6524.351872	130.4870374	1.231586038	1.102987801	0.615793019	2.757469502	0.003549525
45.14%	4.958323354	0.555789723	2.479161677	1.862632409	0.001427587	8878.58117	133.1787176	0.991664671	0.619465553	0.495832335	2.064885177	0.001672431
45.14%	6.978939117	0.735937497	3.489469559	2.453124991	0.0030267	11092.74686	166.3912029	1.395787823	0.768447698	0.697893912	2.561492326	0.003162832

Figure A1 Tensile properties of cellulose stearate film



(A) 55.41% of esterification



(B) 71.38% of esterification

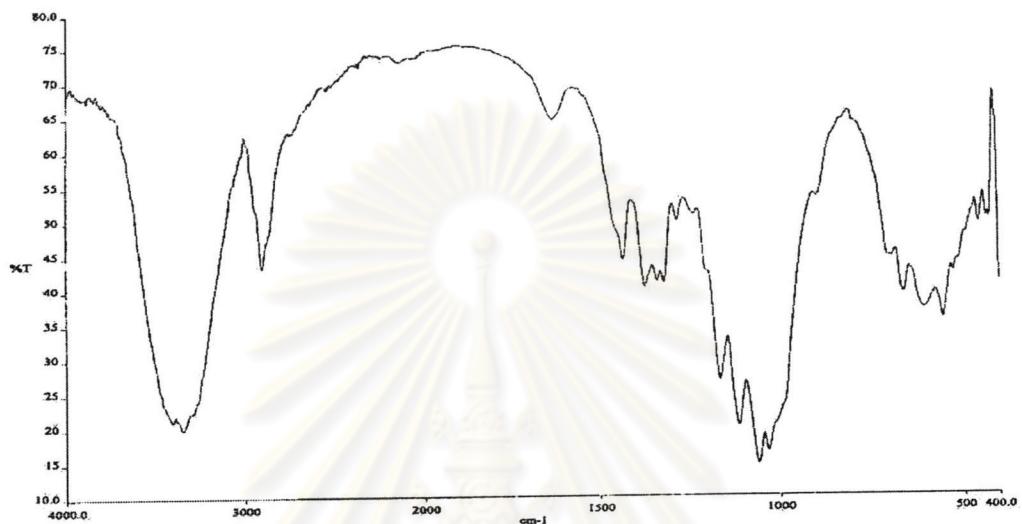
**Fourier Transform Infrared Spectrometer**

Figure A2 FTIR of cellulose

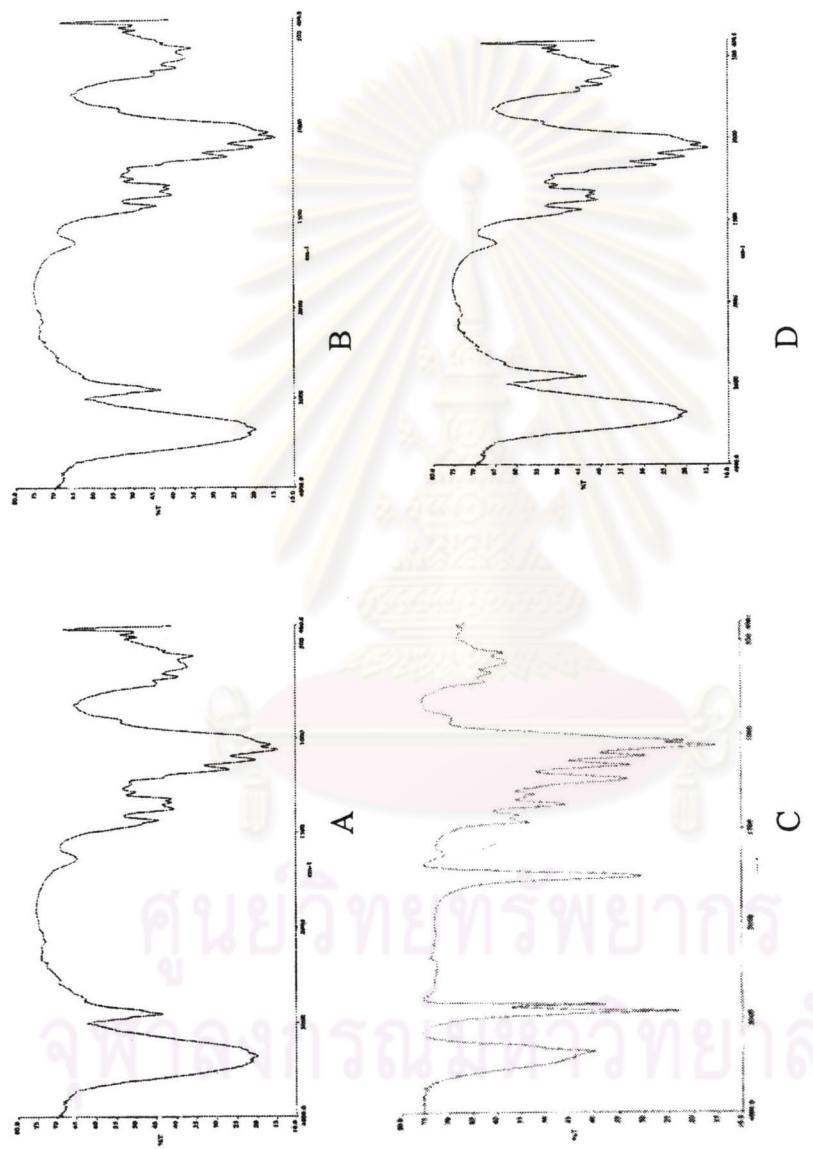


Figure A3 FTIR of Cellulose stearate : 1 eq. of TsCl, 4 eq. pyridine/TsCl at different of eq. of stearic acid  
(A) 0.5 eq. of stearic acid (B) 1 eq. of stearic acid (C) 2 eq. of stearic acid (D) 3 eq. of stearic acid

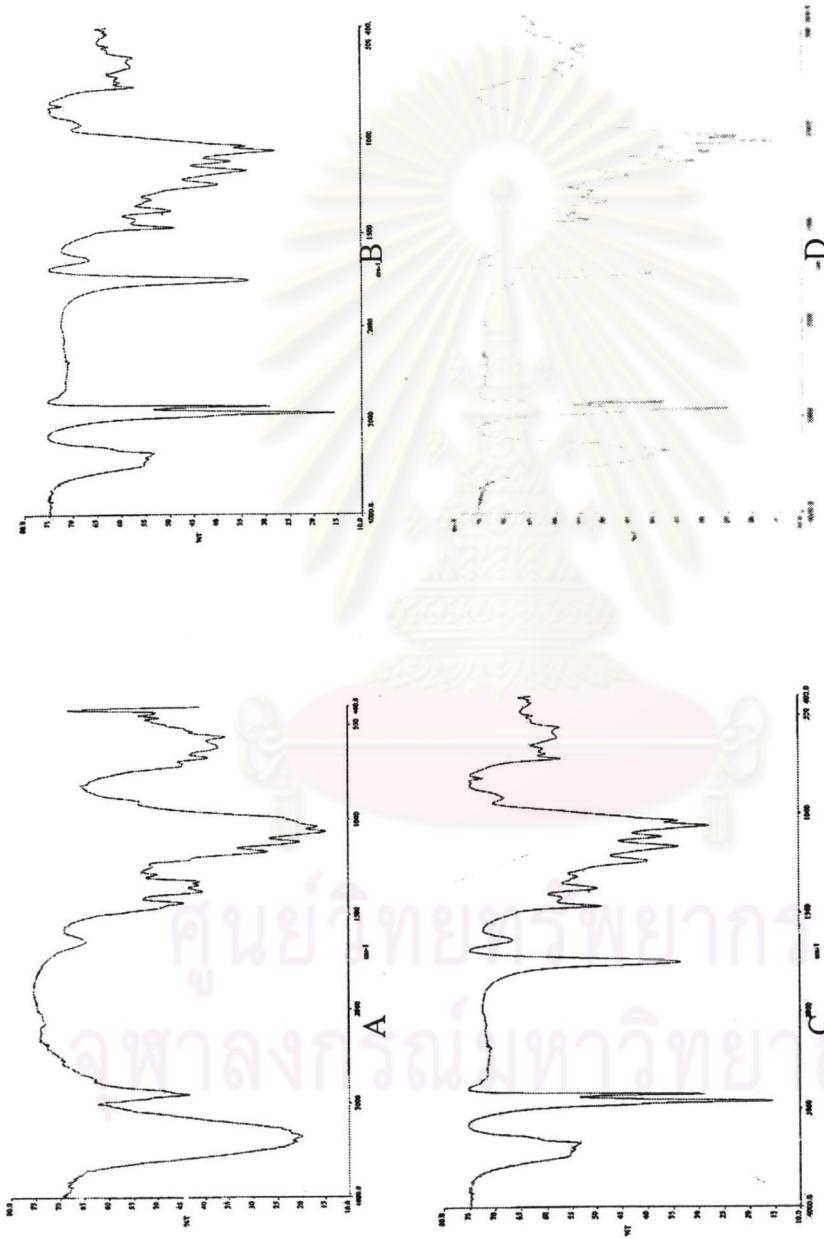


Figure A4 FTIR of Cellulose stearate : 2 eq. of TsCl, 4 eq. pyridine/TsCl at different of eq. of stearic acid (A) 0.5 eq. of stearic acid (B) 1 eq. of stearic acid (C) 2 eq. of stearic acid (D)3 eq. of stearic acid

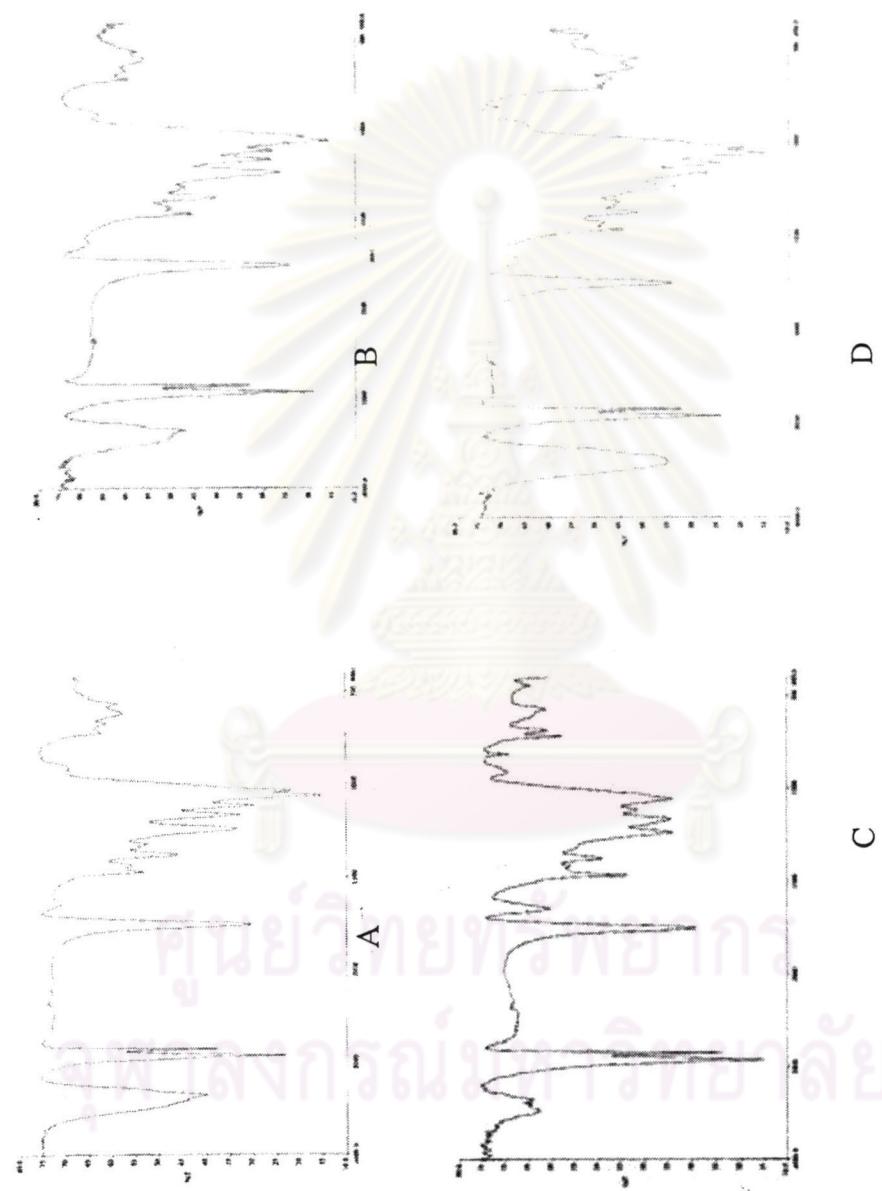


Figure A5 FTIR of Cellulose stearate : 3 eq. of TsCl, 4 eq. pyridine/TsCl at different of eq. of stearic acid  
(A) 0.5 eq. of stearic acid (B) 1 eq. of stearic acid (C) 2 eq. of stearic acid (D) 3 eq. of stearic acid

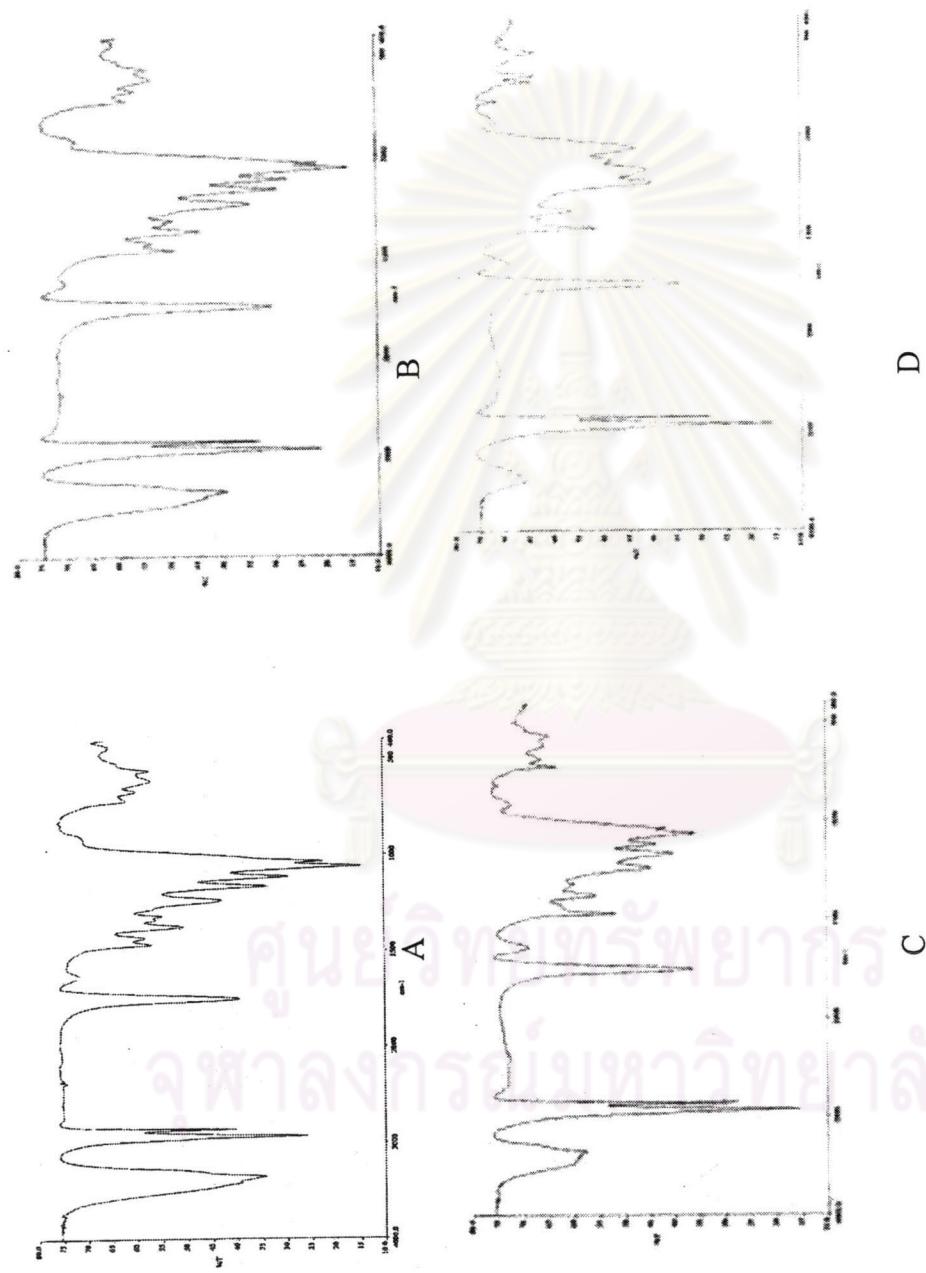


Figure A6 FTIR of Cellulose stearate : 4eq. of TsCl, 4 eq. pyridine/TsCl at different of eq. of stearic acid  
(A) 0.5 eq. of stearic acid (B) 1 eq. of stearic acid (C) 2 eq. of stearic acid (D)3 eq. of stearic acid

### Thermogravimetric Analysis

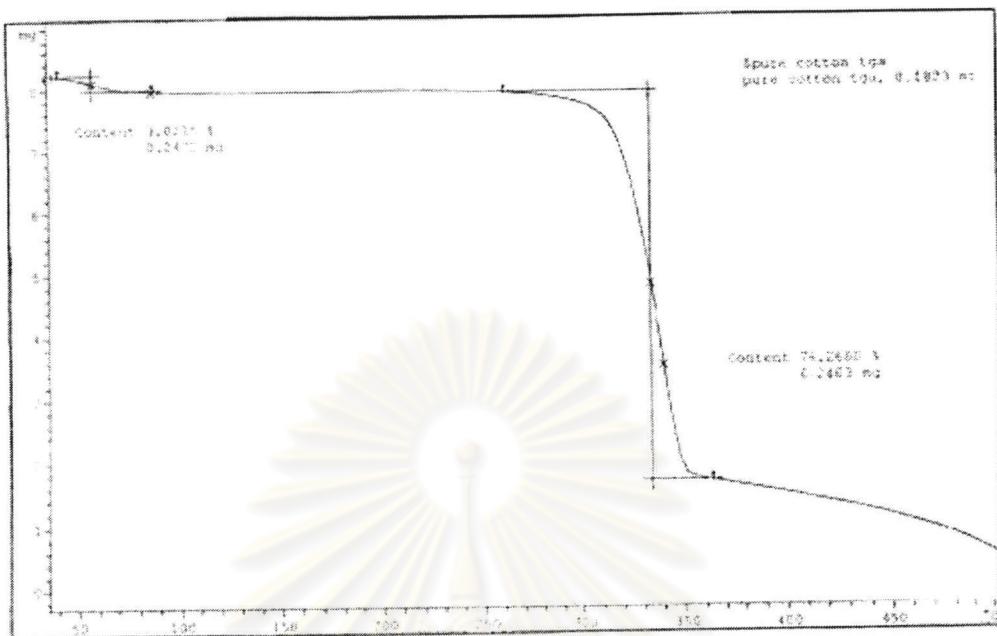


Figure A7 TGA of cotton : heating rate  $10^{\circ}\text{C}/\text{min}$

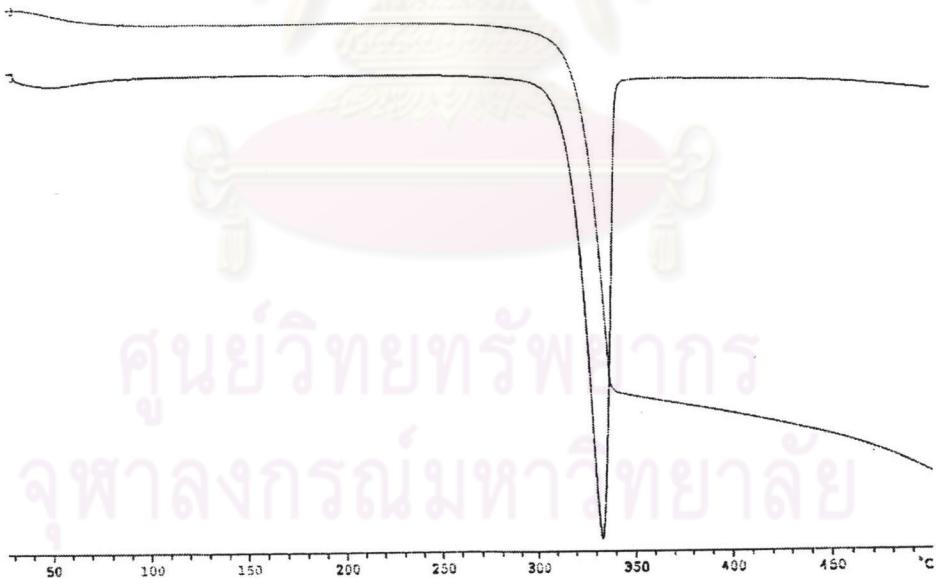


Figure A8 TGA Derivative of cotton

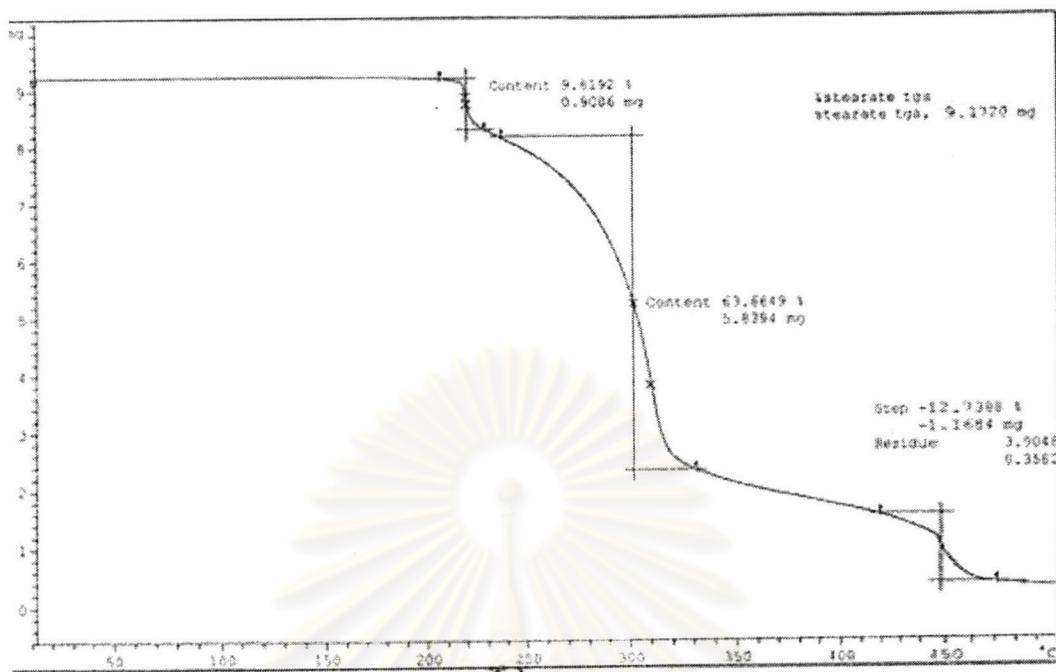


Figure A9 TGA of cellulose stearate: 2eq. of TsCl, 2eq. of stearic acid, 4 eq. pyridine/TsCl at 270 watt for 3.30min (heating rate 10°C/min)

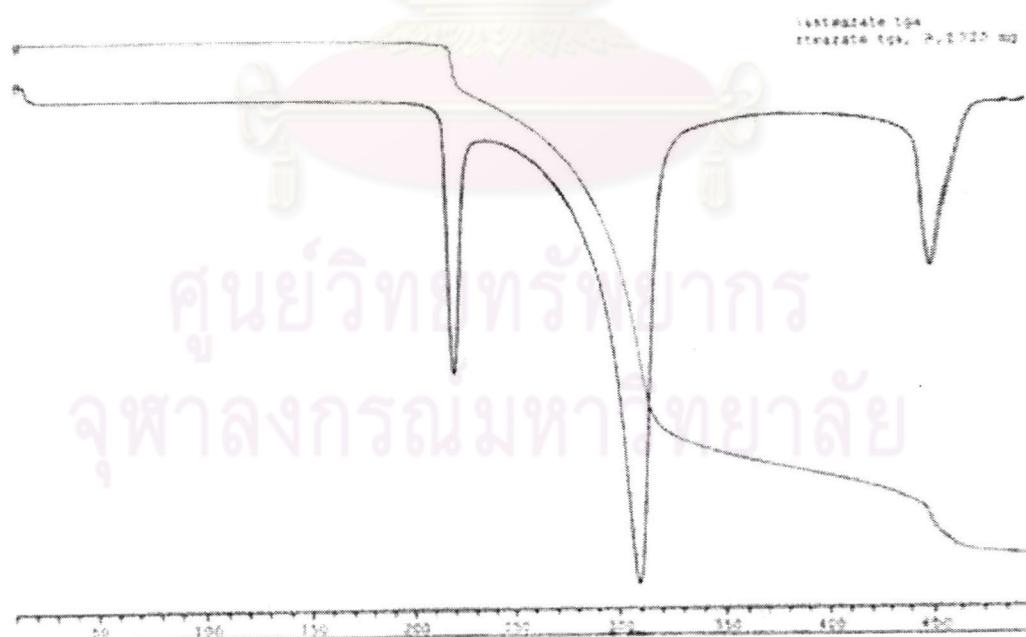


Figure A10 TGA Derivative of cellulose stearate

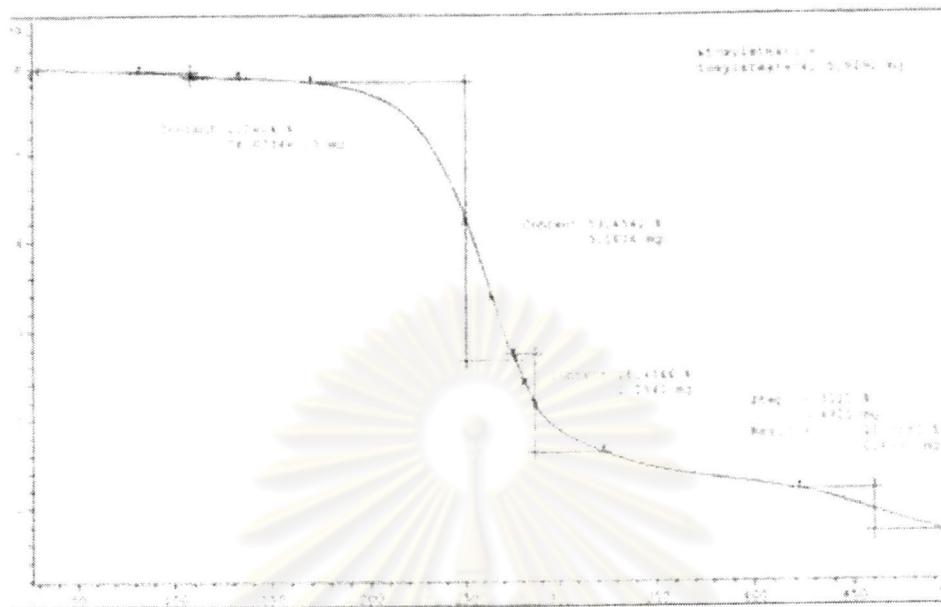


Figure A11 TGA of tosyl cellulose stearate: 4eq. of TsCl, 3eq. of stearic acid, 4 eq. pyridine/TsCl at 270 watt for 3.30min (heating rate 10<sup>0</sup>C/min)

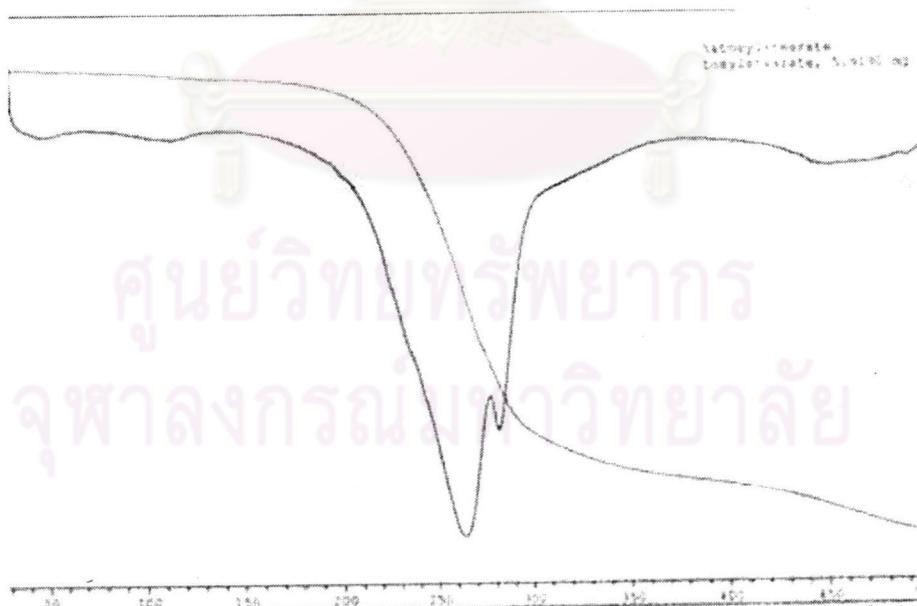


Figure A12 TGA Derivative of Tosyl cellulose stearate

### Differential scanning calorimeter Machine

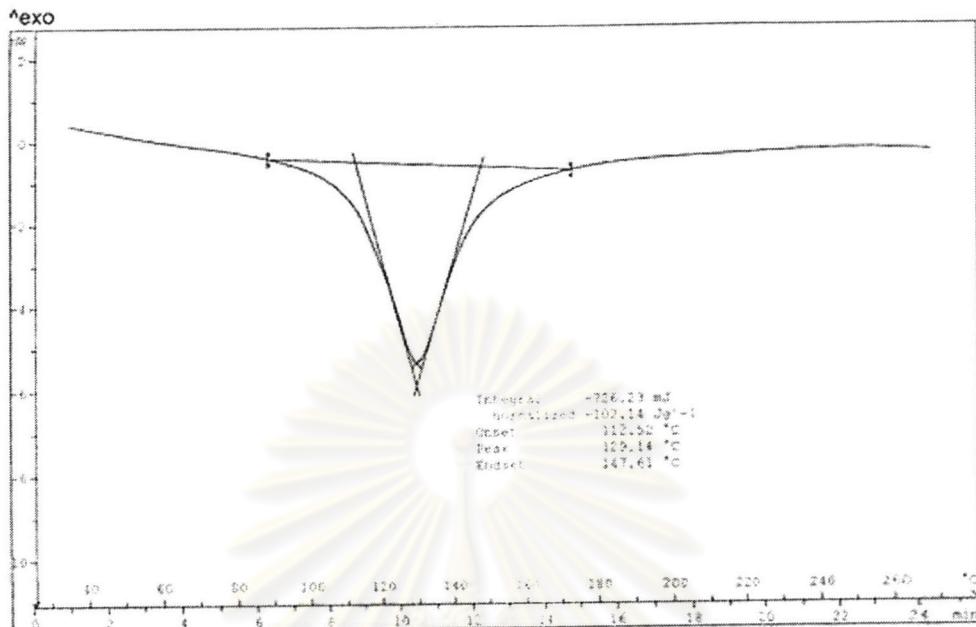


Figure A13 DSC of Cellulose

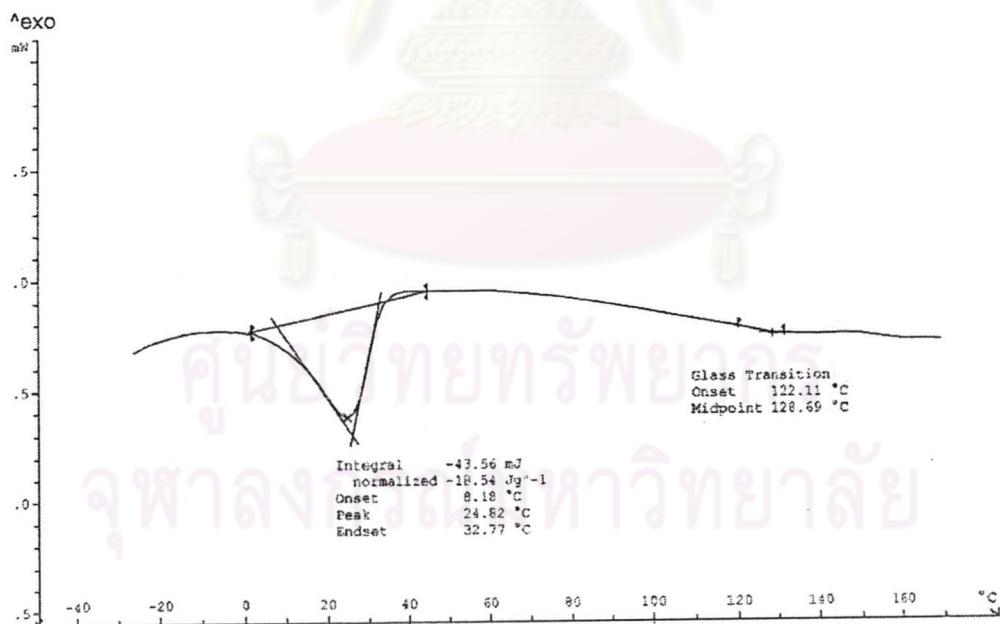


Figure A14 DSC of Cellulose stearate: 2eq. of TsCl, 2eq. of stearic acid, 4 eq. pyridine/TsCl at 270 watt for 3.30min

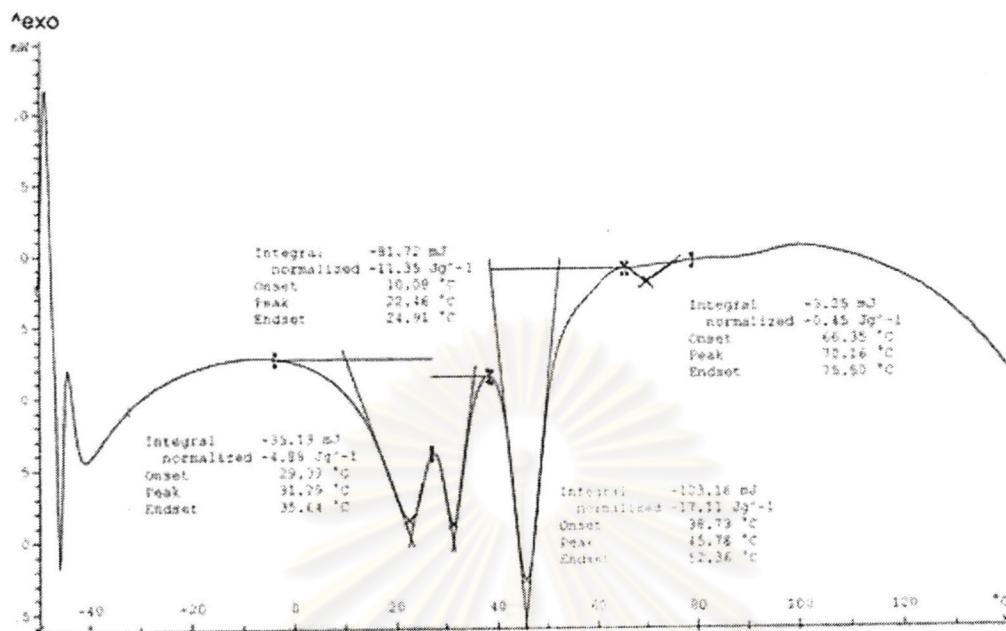
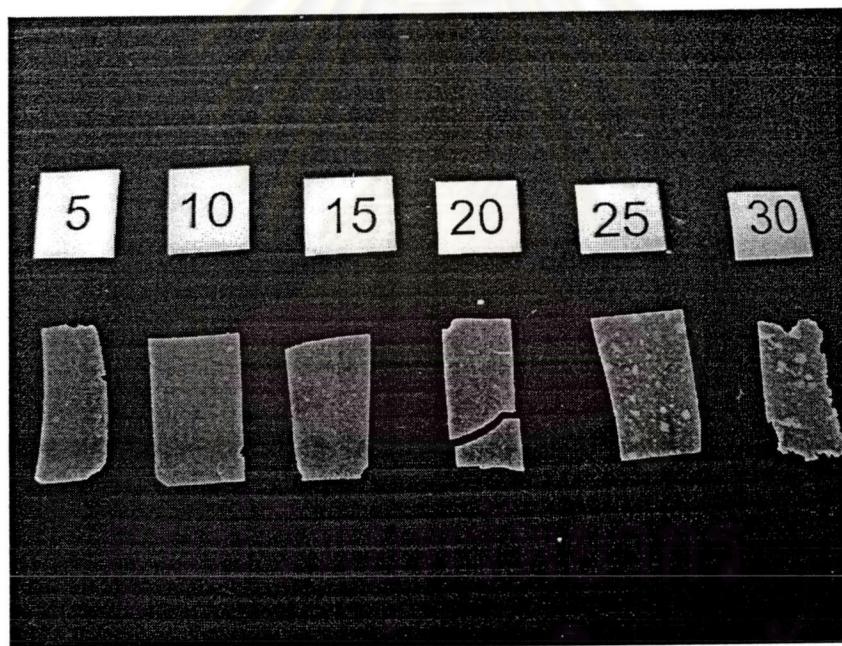


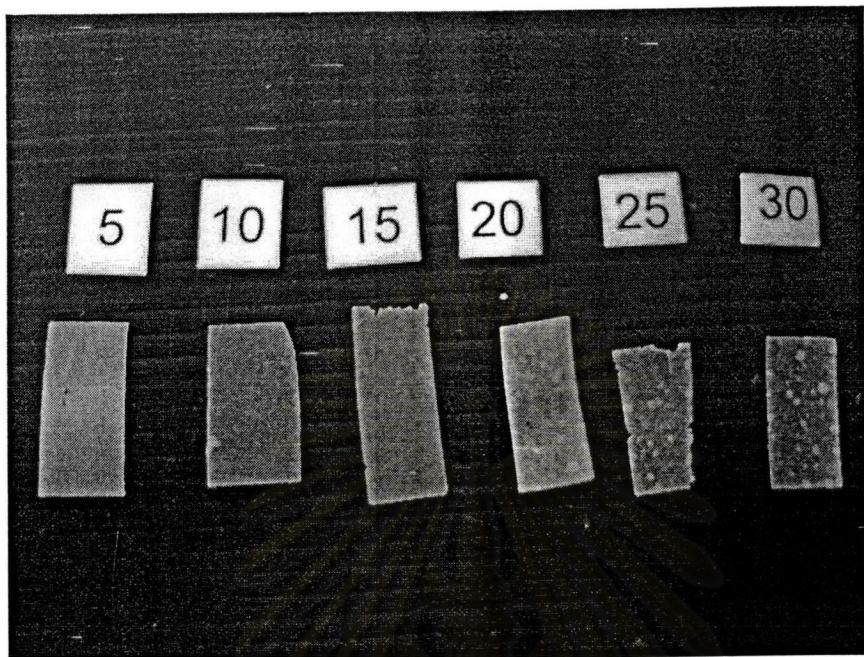
Figure A15 DSC of Tosyl cellulose stearate: 4eq. of TsCl, 3eq. of stearic acid, 4 eq. pyridine/TsCl at 270 watt for 3.30min

**Table A8 Soil bury test of cellulose stearate sample**

	Days	5	10	15	20	25	30
45.14%	Weight before	0.0187	0.0318	0.0322	0.0175	0.0181	0.0171
	Weight after	0.0184	0.0312	0.0306	0.0166	0.0163	0.0145
	%Weight loss	1.604278	1.886792	4.968944	5.142857	9.944751	15.20468
55.41%		0.0508	0.0467	0.035	0.0349	0.0424	0.0415
		0.0499	0.0458	0.033	0.032	0.037	0.0339
		1.771654	1.927195	5.714286	8.309456	12.73585	18.31325
71.38%		0.0465	0.0422	0.0766	0.0397	0.0605	0.043
		0.0456	0.0409	0.0722	0.0355	0.0529	0.0339
		1.935484	3.080569	5.744125	10.57935	12.56198	21.16279

**Soil bury test of cellulose stearate sample**

(A)



(B)



(C)

Figure A16 appearance of film after soil bury test at different time  
(A) 44.14% of esterification (B) 55.41% of esterification (C) 71.38% of esterification

## Appendix B

### 1) Determination of % Yield of esterification

$$\% \text{increase weight} = \frac{W_i - W_f}{W_f} \times 100$$

Where  $W_i$  = Weight of dry sample after terminate reaction

$W_f$  = Weight of dry sample before esterification

### 2) Determination of % Esterification of sample

$$A = \log I_0 / I$$

$$\frac{A_C - A_s}{A_C} = C_S / 100$$

Where  $A_C$  is Absorbance of cellulose

$A_s$  is Absorbance of cellulose stearate sample

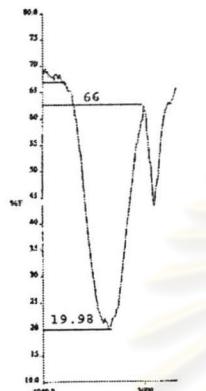
$I$  is % transmittance of OH functional group at maximum

$I_0$  is % transmittance of OH functional group at baseline

$C_S$  is % ester of cellulose sample

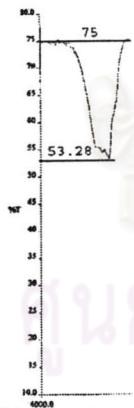
Example

-For cellulose



$$\begin{aligned}\text{Cellulose : } A1 &= \log 66 / 19.98 \\ &= 0.518948452\end{aligned}$$

-For cellulose stearate sample at 155.88 % Yield



Cellulose stearate sample

$$\begin{aligned}A2 &= \log 75 / 53.28 \\ &= 0.148497\end{aligned}$$

Therefore, % Esterification

$$\begin{aligned}(0.52 - 0.15) / 0.52 &= Cs / 100 \\ Cs &= 71.15 \%\end{aligned}$$

### 3) Determination of Water Absorption

$$\% \text{increase weight} = \frac{\text{wet weight} - \text{conditioned weight}}{\text{conditioned weight}} \times 100$$

### 4) Determination of biodegradability

$$\% \text{ Weight loss} = \frac{W_i - W_f}{W_f} \times 100$$

where  $W_i$  is Weight before degradation (g)

$W_f$  is Weight after degradation (g)

## Biography

Mr. Prasit Pattananuwat was born on 13 August 1980 in Bangkok province, Thailand. He received his bachelor degree of Engineering majoring in petrochemical and polymeric material of Engineering and Industrial Technology at Silpakorn University in 2002. After that, he continued doing his master degree at Department of Material Science, Faculty of Science Chulalongkorn University, and obtained his master degree in polymer science and textile technology in September 2004.

