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APPENDICES

Appendix A Weight of As-cast H-chitosan/PLA Blend Films

Blend films of H-chitosan and PLA with different compositions (i.e. the weight ratios between H-chitosan and PLA of 100/0, 80/20, 60/40, 50/50, 40/60, 20/80, and 0/100, respectively) were then prepared by casting a mixture of the solutions in a respective weight ratio on a Teflon dish. The casting was dried at room temperature for one day and later at room temperature in vacuo for another two days. The weight of as-cast blend films casting from chloroform and dichloromethane were listed in table A.1 and A.2, respectively.

Table A.1 Weight of as-cast H-chitosan/PLA blend films cast from chloroform*

H-chitosan/PLA blend composition	Weight (mg)			
	1	2	3	Average
100/0	3.33	3.28	3.32	3.31±0.03
80/20	3.65	3.60	3.76	3.67±0.08
60/40	3.64	3.49	3.77	3.63±0.14
50/50	4.04	3.96	3.63	3.88±0.22
40/60	3.59	3.8	3.68	3.69±0.10
20/80	4.11	4.08	4.19	4.13±0.06
0/100	3.47	3.34	3.68	3.50±0.17

* All of as-cast films prepared from 5 g of solution

Table A.2 Weight of as-cast H-chitosan/PLA blend films cast from dichloromethane*

H-chitosan/PLA blend composition	Weight (mg)			Average
	1	2	3	
100/0	5.32	5.45	5.46	5.41±0.08
80/20	5.85	5.92	5.83	5.87±0.05
60/40	5.77	5.98	5.86	5.87±0.10
50/50	6.16	6.08	6.18	6.14±0.05
40/60	5.84	5.98	5.98	5.94±0.08
20/80	5.92	5.96	5.88	5.92±0.04
0/100	7.56	7.64	7.44	7.55±0.10

* All of as-cast films prepared from 5 g of solution

Appendix B Percentages of Weight Loss after Etching of As-cast H-chitosan/PLA Blend Films

Prior to observation under SEM, the blend films were either etched with cyclohexane or concentrated acetic acid solution for two minutes at room temperature in order to remove H-chitosan or PLA, respectively. The weight losses of as-cast blend films were carried out by measuring the weight of the films after etching in etched solvent for two minutes at room temperature in comparison with the dry weight of the films prior to etching. The percentage of weight loss was determined according to the following relationship:

$$\text{Percentage of weight loss (\%)} = [W_d - W_e / W_d] \times 100, \quad (1)$$

where W_d and W_e represent the weight of the films before and after etching.

The percentages of weight loss of as-cast blend films casting from chloroform and dichloromethane were listed in table B.1 and B.2, respectively.

Table B.1 Percentages of weight loss after etching of as-cast H-chitosan/PLA blend films cast from chloroform

H-chitosan/PLA blend composition	Weight (mg)						% Weight Loss			
	Before Etching			After Etching						
	1	2	3	1	2	3	1	2	3	Average
80/20c	3.1	3.2	3.3	2.7	2.6	2.9	12.90	18.75	12.12	14.59±3.62
60/40c	1.2	1.6	1.7	0.7	0.9	0.8	41.67	43.75	52.94	46.12±6.00
50/50c	1.9	2.3	2.8	0.8	1.2	1.5	57.89	47.83	46.43	50.72±6.26
50/50a	1.7	1.7	1.6	0.6	0.8	0.8	64.71	52.94	50.00	55.88±7.78
40/60a	1.4	1.9	2.3	0.8	1	1.5	42.86	47.37	34.78	41.67±6.38
20/80a	2.9	2.9	2.6	2.2	2.5	2.2	24.14	13.79	15.38	17.77±5.57

a = etched by concentrated acetic acid solution

c = etched by cyclohexane

Table B.2 Percentages of weight loss after etching of as-cast H-chitosan/PLA blend films cast from dichloromethane

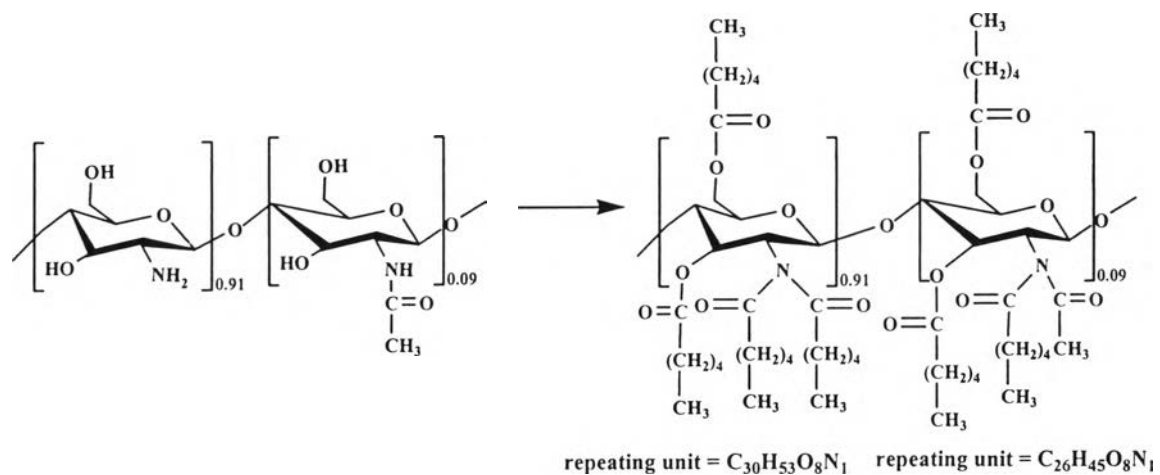
H-chitosan/PLA blend composition	Weight (mg)						% Weight Loss			
	Before Etching			After Etching			1	2	3	Average
	1	2	3	1	2	3				
80/20c	4.5	5.1	5.3	4	4.2	4.2	11.11	17.65	20.75	16.50±4.92
60/40c	6.1	6.7	7.4	3.5	4.6	4.4	42.62	31.34	40.54	38.17±6.00
50/50c	3.8	4.1	4.3	2	2.4	2.2	47.37	41.46	48.84	45.89±3.90
50/50a	3.3	3.6	3.6	1.8	1.7	1.5	45.45	52.78	58.33	52.19±6.46
40/60a	4.9	4.9	5.6	2.9	2.7	3	40.82	44.90	46.43	44.05±2.90
20/80a	5.9	6.3	6.9	5	5.1	5.6	15.25	19.05	18.84	17.71±2.13

a = etched by concentrated acetic acid solution

c = etched by cyclohexane

Appendix C Degree of Substitution of H-Chitosan

The calculated value of %C, %H and %N of H-chitosan with fully substitution were calculated according the following:



Chitosan %DD = 91

Fully substituted H-chitosan

Figure C.1 A synthesis reaction of fully substitution H-chitosan

For deacetylated unit, $C_{30}H_{51}O_8N_1$

$$\text{Atomic weight} = (30 \times 12) + (51 \times 1) + (8 \times 16) + (14 \times 1) = 553$$

$$\% C = (30 \times 12) / 553 = 65.10$$

$$\% H = (51 \times 1) / 553 = 9.22$$

$$\% N = (14 \times 1) / 553 = 2.53$$

For acetylated unit, $C_{26}H_{43}O_8N_1$

$$\text{Atomic weight} = (26 \times 12) + (43 \times 1) + (8 \times 16) + (14 \times 1) = 497$$

$$\% C = (26 \times 12) / 497 = 62.78$$

$$\% H = (43 \times 1) / 497 = 8.65$$

$$\% N = (14 \times 1) / 497 = 2.82$$

H-chitosan was prepared by reaction of chitosan with % DD of 91, it means that chitosan chains have deacetylated group about 91 groups per 100 repeating units and remain acetylated group about 9 groups. Then

$$\% C_{\text{total}} = [\%C_{\text{deacetylated group}} \times 0.91] + [\%C_{\text{acetylated group}} \times 0.09]$$

$$\% H_{\text{total}} = [\%H_{\text{deacetylated group}} \times 0.91] + [\%H_{\text{acetylated group}} \times 0.09]$$

$$\% N_{\text{total}} = [\%N_{\text{deacetylated group}} \times 0.91] + [\%N_{\text{acetylated group}} \times 0.09]$$

so

$$\% C_{\text{total}} = [65.10 \times 0.91] + [62.78 \times 0.09] = 64.89$$

$$\% H_{\text{total}} = [9.22 \times 0.91] + [8.65 \times 0.09] = 9.17$$

$$\% N_{\text{total}} = [2.53 \times 0.91] + [2.82 \times 0.09] = 2.56$$

$$\%C/N = 64.89/2.56 = 25.34$$

Fully substituted H-chitosan contains 4 substituted positions, which is correlated to 25.41 percentage ratio of carbon to nitrogen atom. Then the degree of substitution is calculated by correlation of percentage ratio of carbon to nitrogen atom to the number of substituted group.

The results of elemental analysis of H-chitosan with different degrees of substitution are listed in table C.1.

Table C.1 Elemental analyses of H-chitosan

Derivatives	C%	H%	N%	C/N	%DS
Calculated values	64.89	9.17	2.56	25.34	4.00
H1-chitosan	52.55	8.57	4.02	13.06	2.06
H2-chitosan	66.09	9.41	2.76	23.96	3.78
H3-chitosan	66.84	10.24	2.74	24.77	3.91
H4-chitosan	66.48	10.01	2.68	24.85	3.92

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Publications:

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2. Peesan, M., Supaphol, P., and Rujiravanit, R., (2004, August 31 – September 3) Preparation and Characterization of Hexanoyl Chitosan/Poly(lactide) Blend Films. Oral presented at 6th International Conference of the European Chitin Society. Poznan, Poland.
3. Preechawong, D., Peesan, M., Rujiravanit, R., and Supaphol, P., (2003, November 24-27) Preparation and Characterization of Starch-Based Composite Foams. Poster presented at 8th Pacific Polymer Conference. Bangkok, Thailand.
4. Peesan, M., Rujiravanit, R., and Supaphol, P., (2003, July 23-25) Preparation and Characterization of Blend Films from Beta-Chitin and Poly(vinyl alcohol). Poster presented at An International Conference on Advances in Petrochemicals and Polymers in the New Millennium. Bangkok, Thailand.
5. Peesan, M., Rujiravanit, R. and Supaphol, P., (2002, March 13-15) Physical Properties of β -Chitin/Poly(vinyl alcohol) Blend Films. Oral presented at Proceedings of the 5th Asia Pacific Chitin and Chitosan Symposium & Exhibition. Bangkok, Thailand.