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## APPENDICES

### Appendix A Some Calculations for Layered Clays

#### Cation Exchange Capacity (CEC)

2.0 grams of purified clay were dispersed in 300 mL. The dispersion was stirred until a uniform dispersion is obtained. The pH value was adjusted to 2.5 to 3.8 by sulfuric acid. The suspension was continued stirring for 15 min. Methylene blue solution (0.01 N) was added slowly by buret into the clay slurry while maintains a constants stirring. A drop of the suspended solution was place on a filter paper for end point detection. The end point was indicated by a formation of light blue halo around the drop. The methylene was continued to add at 1.0 mL increasing until the end point was reached. After the end point was reached, the solution was stirred of 2 min and retest for the end point.

The calculation the methylene blue index can calculated from below equation:

$$MBI = \frac{E * V * 100}{W}$$

where:

MBI = methylene blue index for the clay in meq/100 g clay

E = concentration of the methylene blue (in milliequivalents of methylene blue per milliter)

V = amount of methylene blue (in milliliter) required for the titration

W = weight of dry clay in gram

#### Swelling Index of layered silicate

The swelling index of layered silicate clays was measured by the following method (D 5890-02): 2 g of the clay was poured into 100 ml of distilled water (or *n*-dodecane) in a mass cylinder. After 24 h, the apparent volumes of the swelling clays were measured.

Chemical formula of Organomodified LDHs

LDH-NO<sub>3</sub>

Formula = Mg<sub>0.66</sub>Al<sub>0.34</sub>(OH)<sub>2</sub>(NO<sub>3</sub>)<sub>0.34</sub>•xH<sub>2</sub>O, calculation x value

Amount of water = 9.3% (from TGA analysis)

$$x \text{ (mol of water)} = ((9.3/18)/100)/(80.298+18x)$$

$$100x = 41.49 + 9.3x$$

$$x = 0.46$$

So molecular of LDH-NO<sub>3</sub> = Mg<sub>0.66</sub>Al<sub>0.34</sub>(OH)<sub>2</sub>(NO<sub>3</sub>)<sub>0.34</sub>•0.46H<sub>2</sub>O

LDH -C8:

Sodium 2-ethylhexyl sul fate (C6, branch C2 )

MW = 232 g/mol , but real MW inside LDH = 232-23 = 209 g/mol

Formula = Mg<sub>0.66</sub>Al<sub>0.34</sub>(OH)<sub>2</sub>(CO<sub>3</sub><sup>2-</sup>)<sub>z</sub>(LDH-C8)<sub>y</sub>•xH<sub>2</sub>O, calculation x, y, and z

Amount of water = 8.1%

Residue = 40.8%

**Note:** from TGA analysis the residue is = Mg<sub>0.66</sub>Al<sub>0.34</sub>O<sub>1.17</sub>, MW = 43.34 g/mol

$$MW_{\text{tot}} : 100 = 43.34 : 40.8$$

$$MW_{\text{tot}} = 106.22 \text{ g/mol} \rightarrow \text{LDH-C8}$$

Calculate x:

$$x = ((8.1/18)/100)/106.22$$

$$= 0.48$$

Calculate y:

$$106.22 = 59.22 + (60(0.34-y)/2) + 209y + (0.48)18$$

$$y = 0.157$$

Calculate z:

$$2z + y = 0.34$$

$$z = 0.092$$

\*\*\*LDH-C8 formula = Mg<sub>0.66</sub>Al<sub>0.34</sub>(OH)<sub>2</sub>(CO<sub>3</sub><sup>2-</sup>)<sub>0.092</sub>(LDH-C8)<sub>0.157</sub>•0.48H<sub>2</sub>O

Real MW of LDH-C8 = 106.19 g/mol



LDH-C12

Sodium dodecyl sulfate (C12) -SDS

MW = 288 g/mol , but real MW inside LDH = 288-23 = 265 g/mol

Formula =  $Mg_{0.66}Al_{0.34}(OH)_2(CO_3^{2-})_z(LDH-DS)_y \cdot xH_2O$ ,

calculation x, y, and z

Amount of water = 8.8%

Residue = 38.8%

**Note:** from TGA analysis the residue is =  $Mg_{0.66}Al_{0.34}O_{1.17}$ , MW = 43.34 g/mol

$$MW_{tot} : 100 = 43.34 : 38.8$$

$$MW_{tot} = 111.41 \text{ g/mol} \rightarrow \text{LDH-DS}$$

Calculate x:

$$\begin{aligned} x &= ((8.8/18)/100)/111.41 \\ &= 0.54 \end{aligned}$$

Calculate y:

$$\begin{aligned} 111.41 &= 59.22 + (60(0.34 - y)/2) + 265y + (0.54)18 \\ y &= 0.137 \end{aligned}$$

Calculate z:

$$\begin{aligned} \text{from } 2z + y &= 0.34 \\ z &= 0.10 \end{aligned}$$

\*\*\*LDH-C12 formula =  $Mg_{0.66}Al_{0.34}(OH)_2(CO_3^{2-})_{0.1}(LDH-C8)_{0.137} \cdot 0.54H_2O$ 

Real MW of LDH-DS = 111.25 g/mol

LDH -C20

Sodium eicosyl sul fate (C20)

MW = 379 g/mol, but real MW inside LDH = 379-23 = 356 g/mol

Formula =  $Mg_{0.66}Al_{0.34}(OH)_2(CO_3^{2-})_z(LDH-C20)_y \cdot xH_2O$ ,

calculation x, y, and z

Amount of water = 7.6%

Residue = 28.6%

**Note:** from TGA analysis the residue is =  $Mg_{0.66}Al_{0.34}O_{1.17}$ , MW = 43.34 g/mol

$$MW_{\text{tot}} : 100 = 43.34 : 28.6$$

$$MW_{\text{tot}} = 151.54 \text{ g/mol} \rightarrow \text{LDH-C20}$$

Calculate x:

$$\begin{aligned} x &= ((7.6/18)/100)/151.54 \\ &= 0.64 \end{aligned}$$

Calculate y:

$$\begin{aligned} 151.54 &= 59.22 + (60(0.34 - y)/2) + 356y + (0.64)18 \\ y &= 0.216 \end{aligned}$$

Calculate z:

$$\text{from } 2z + y = 0.34$$

$$z = 0.062$$

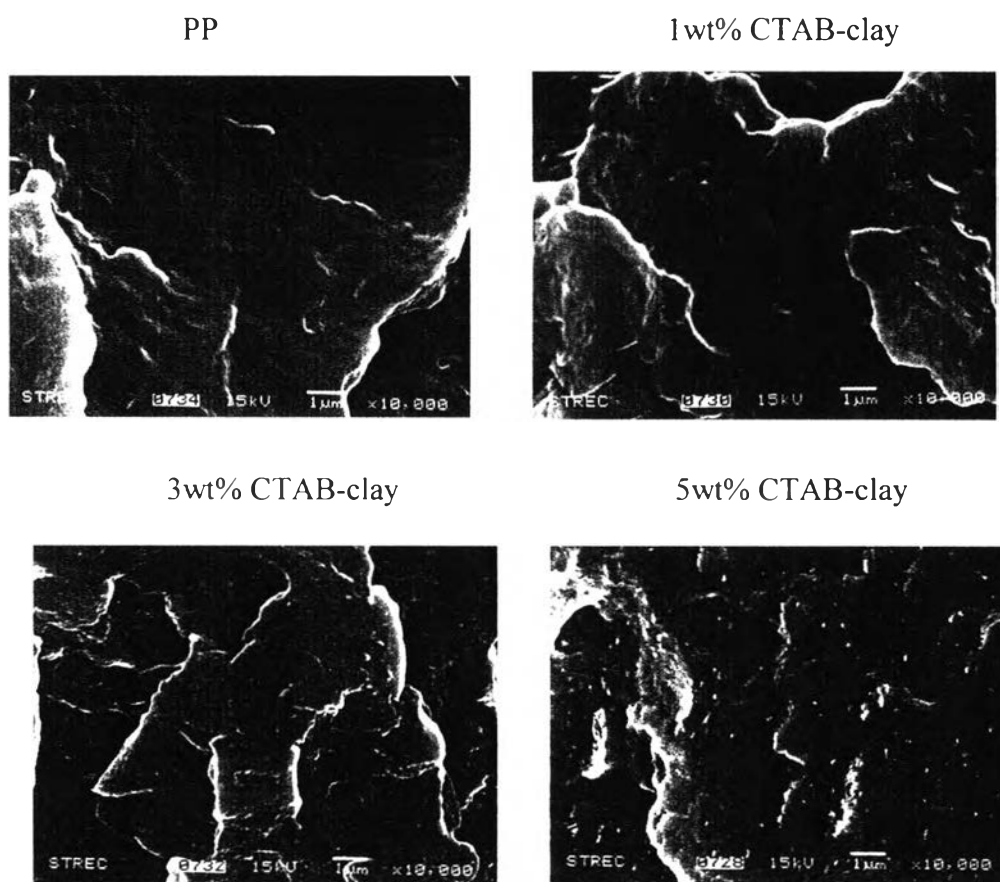
$$***\text{Real formula} = \text{Mg}_{0.66}\text{Al}_{0.34}(\text{OH})_2(\text{CO}_3^{2-})_{0.062}(\text{LDH-C8})_{0.216} \cdot 0.64\text{H}_2\text{O}$$

$$\text{Real MW of LDH-DS} = 151.36 \text{ g/mol}$$

## Appendix B Supplementary Results of Chapter IV

### SEM image of PP/OBTN nanocomposites

The dispersion of clay particles and the extent of intercalated/exfoliated structure of PP/clay nanocomposites were characterized by electron microscopy (SEM) at 10,000 magnifications, shown in Figure B1.



**Figure B1** SEM images of PP/CTAB-clay nanocomposites at magnifications of X10000.

### Appendix C Supplementary Results of Chapter VII

**Table C1** Assignment of various XRD reflections for LDH-NO<sub>3</sub> and Organo-LDHs

Materials	reflections in $\langle 00l \rangle$ series					
	$\langle 003 \rangle$		$\langle 006 \rangle$		$\langle 009 \rangle$	
	$2\theta$ (deg)	$d$ (nm)	$2\theta$ (deg)	$d$ (nm)	$2\theta$ (deg)	$d$ (nm)
LDH-CO <sub>3</sub>	11.69	0.76	23.40	0.38	34.62	0.26
LDH-NO <sub>3</sub>	9.91	0.89	19.88	0.45	34.62	0.26
LDH-NO <sub>3</sub> _C8	4.16	2.12	8.46	1.04	12.55	0.74
LDH-NO <sub>3</sub> _C12	3.65	2.42	7.25	1.22	10.85	0.81
LDH-NO <sub>3</sub> _C20	2.72	3.25	5.49	1.61	8.27	1.07

**Table C2** Assignment of various XRD reflections obtained for LDPE/modified LDHs composites

Sample	reflections in $\langle 00l \rangle$ series					
	$\langle 003 \rangle$		$\langle 006 \rangle$		$\langle 009 \rangle$	
	$2\theta$ (deg)	$d$ (nm)	$2\theta$ (deg)	$d$ (nm)	$2\theta$ (deg)	$d$ (nm)
LDH-NO <sub>3</sub>	9.91	0.89	19.88	0.45	34.62	0.26
LDPE-C8	3.89	2.27	8.10	1.09	ND	ND
LDPE_DS	3.24	2.72	6.70	1.32	9.39	0.94
LDPE_C20	2.32	3.80	4.82	1.83	7.39	1.20

**Table C3** Assignment of FTIR bands in LDH-NO<sub>3</sub> and Organomodified LDHs

Materials	Band position (cm <sup>-1</sup> )	Types of Vibration
LDH-NO <sub>3</sub>	3468	$\nu_{\text{-OH}}$ (OH of LDH or co-intercalated)
	1384	$\nu_{\text{-NO}_3}$ (asymmetric)
	680	Al-OH (translation mode)
	550	Mg-OH (translation mode)
LDH-NO <sub>3</sub> _C8	3479	$\nu_{\text{-OH}}$ (OH of LDH or co-intercalated)
	2919, 2852	$\nu_{\text{-CH}_2}$
	1219	$\nu_{\text{S=O}}$ (symmetric)
	1065	$\nu_{\text{S=O}}$ (asymmetric)
	630	$\nu_{\text{C-S}}$
	671, 818, 1371 and 1468	Different vibration modes of CO <sub>3</sub> <sup>2-</sup>
LDH-NO <sub>3</sub> _DS	3479	$\nu_{\text{-OH}}$ (OH of LDH or co-intercalated)
	2919, 2852	$\nu_{\text{-CH}_2}$
	1217	$\nu_{\text{S=O}}$ (symmetric)
	1066	$\nu_{\text{S=O}}$ (asymmetric)
	630	$\nu_{\text{C-S}}$
	678, 816, 1370 and 1470	Different vibration modes of CO <sub>3</sub> <sup>2-</sup>
LDH-NO <sub>3</sub> _C20	3479	$\nu_{\text{-OH}}$ (cointercalated water molecules)
	2919, 2851	$\nu_{\text{-CH}_2}$
	1219	$\nu_{\text{S=O}}$ (symmetric)
	1064	$\nu_{\text{S=O}}$ (asymmetric)
	630	$\nu_{\text{C-S}}$
	680, 820, 1371 and 1470	Different vibration modes of CO <sub>3</sub> <sup>2-</sup>

**Table C4** Designing formula for preparing LDPE/LDHs-nanocomposites

Sample	LDPE (g)	PE-g-MA (g)	LDH (g)
LDPE/LDPE-g-MA	19.5	0.5	-
LDH-C8 composite	19.5	0.5	0.5
LDH-DS composite	19.5	0.5	0.5
LDH-C20 composite	19.7	0.3	0.3

Processing conditions in Babender:

Temperature = 170 °C

Speed = 80 rpm

Residence time = 20 min.

## CURRICULUM VITAE

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**Publications:**

1. Muksing, N., Nithitanakul, M., Grady, BP., and Magaraphan, Rathanawan., (2008) Melt rheology and extrudate swell of organobentonite-filled polypropylene nanocomposites. Polymer Testing, 27, 470-479.
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**Proceedings:**

1. Muksing, N., Coiai, S., Conzatti, L., Passaglia, E., Magaraphan, R., and Ciardelli, F. (2010, April 7-10). Evidence of anionic guests intercalated layered double hydroxide host on morphology and thermal properties of LDPE based nanocomposites. Paper present at The POLYCHAR18–World Forum on Advanced Materials, Siegen, Germany (Poster prize).
2. Muksing, N., Nithitanakul, M., Grady, BP., and Magaraphan, R. (2008, June 15-19) Melt rheology and extrudate swell of organobentonite-filled polypropylene nanocomposites. Proceedings of the 24<sup>th</sup> Polymer Processing Society Annual Meeting (PPS-24), Salerno, Italy.
3. Muksing, N., Nithitanakul, M., and Magaraphan, R. (2006, October 10-13) Effect of clay contents on morphology, thermal and mechanical properties of polypropylene nanocomposites. Proceedings of the IUPAC International Symposium on Advanced Polymers for Emerging Technologies (The 30<sup>th</sup> PSK), Busan, Korea.
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**Presentations:**

1. Muksing, N., Coiai, S., Conzatti, L., Passaglia, E., Magaraphan, R., and Ciardelli, F. (2010, April 7-10). Evidence of anionic guests intercalated layered double hydroxide host on morphology and thermal properties of LDPE based nanocomposites. Paper present at The POLYCHAR18–World Forum on Advanced Materials, Siegen, Germany (Poster prize).
2. Muksing, N., Nithitanakul, M., Grady, BP., and Magaraphan, R. (2008, June 15-19) Melt rheology and extrudate swell of organobentonite-filled polypropylene nanocomposites. Paper present at The 24<sup>th</sup> Polymer Processing Society Annual Meeting (PPS-24), Salerno, Italy.
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5. Muksing, N., Jirakittidul, K., Phandee, A., Leungsuklerk, S., Nithitanakul, M., Manuspiya, M., and Magaraphan, R. (2005, December 6-7) Modification of the local clay used for polymer nanocomposites. Proceedings of the First Mathematics and Physical Science Graduate Congress, Bangkok, Thailand.