Chemical and Physical Interaction in Polyesters Systems

國立高雄大學 化學工程及材料工程學系

蘇進成

台灣大學高分子所 專題討論課程專用

『高雄』有哪些大學？

I>. 高教體系

國立大學：

國立高雄大學

國立高雄師範大學

國立中山大學

私立大學：

高雄醫學大學

…

II>. 技職體系

國立大學：

國立高雄應用科大

國立高雄第一科大

國立高雄海洋科大

國立高雄餐飲學院

私立大學：

…

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高分大學 專題討論課程專用

Introduction

Compatibility in polymer blends

I>. **Reactive compatibilization**

II>. Addition of block and graft copolymers

III>. Addition of low molecular weight coupling agents

IV>. Utilization of non-bonding specific interactions

Reactive Polymer Blending

Reaction type	Reactive group	Co-reactive group	Remarks
Amidation	Carboxylic acid	Amine	Addition/substitution
Imidation	Anhydride	Amine	
Esterification	Carboxylic acid And anhydride	Hydroxyl	
Concerted addition	Maleate and Man	Double bound	
Urea formation	Carbodiimide	Carboxylic acid	
Urethane formation	Isocyanate	Hydroxyl	
Substitution	Amine	Hydroxyl, halide	
Ester interchange	Ester	Ester	Interchange reaction
Transterification	Ester	Hydroxyl/phenol	
Amide-ester Exchange	Amide	Ester	
Aminolysis	Ester	Amine	
Amide interchange	Amide	Amide	
Acidolysis	Ester	Carboxylic acid	
Ring-opening reaction	Epoxide	Carboxylic acid, Man, Hydroxyl, amine	Ring-opening reaction
Ring-opening reaction	Oxazoline	Carboxylic acid, Man, halide, amine	
Ring-opening reaction	Lactam	Amine	
Ionic bonding	Acid	Pyridine, amine Imidazole	Ionic bonding
Ionic bonding	Ionomer	Iomomer	

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Compatibility in Blends of Poly(ether imide) and Polyesters

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Introduction

- polyesters: *semicrystalline polymers*
  mechanical characteristics

  poly(ether imide) (PEI): *thermal stability*
  toughness, high T_g…

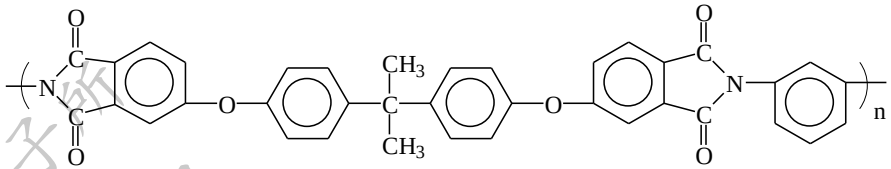
Ex. poly(ethylene terephthalate) (PET)/PEI
poly(trimethylene terephthalate) (PTT)/PEI
poly(butylenes terephthalate) (PBT)/PEI

- Transesterification in blends of Poly(ethylene naphthalate)/aryl polyester

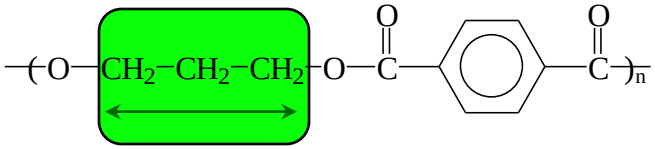
Ex. PEN/PET, PEN/PBT, PC/PET …

Materials

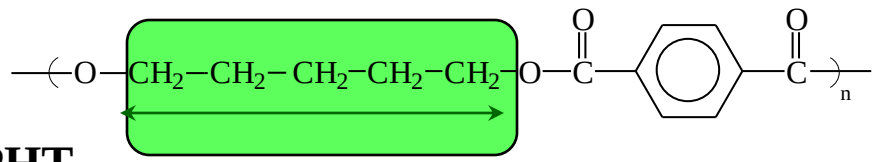
- **Poly(ether imide) PEI**
  Polysciences, Inc., USA
  M_w=30000 g mole^{-1} T_g=215.6℃



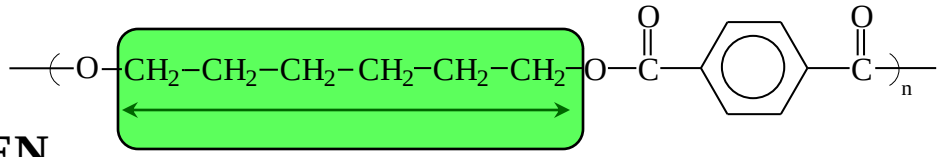
- **Poly(trimethylene terephthalate) PTT**
  ITRI.
  T_g=42.8℃，T_m=228.5℃



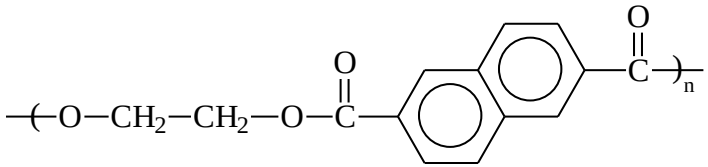
- **Poly(pentamethylene terephthalate) PPT**
  M_w=16600 g mole^{-1} PDI=1.54
  T_g=8.1℃，T_m=129.7℃



- **Poly(hexamethylene terephthalate) PHT**
  M_w=13800 g mole^{-1} PDI=2.06
  T_g= -2℃， T_m =145.7℃



- **Poly(ethylene 2,6-naphthalate) PEN**
  Aldrich Inc.
  T_g =116 ℃，T_m=268℃



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Outline

Part I

PPT/PEI & PHT/PEI binary blends

- Compatibility
- Polymer-polymer interaction parameter

Part II

PEN/PPT/PEI & PEN/PTT/PEI ternary blends

- Compatibility
- Transesterification reaction

Part III

ENTT/PEI blends
ENPT/PEI blends
ENPT/PPT/PEI blends

- Compatibility

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Part I Polyesters and Poly(ether imide) binary blends

- **PPT/PEI & PHT/PEI binary blends**

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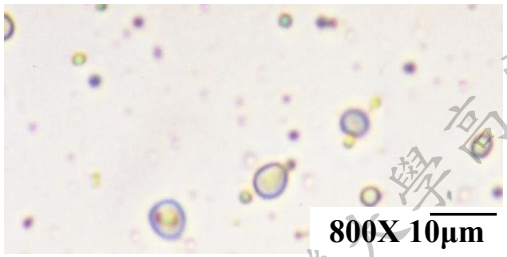
PPT/PEI binary blends

❖POM graphs of co-precipitated PPT/PEI blends



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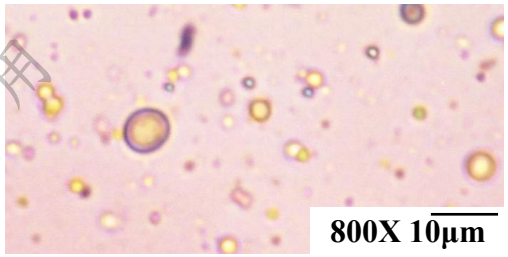

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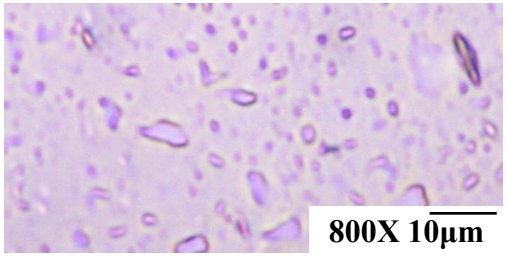

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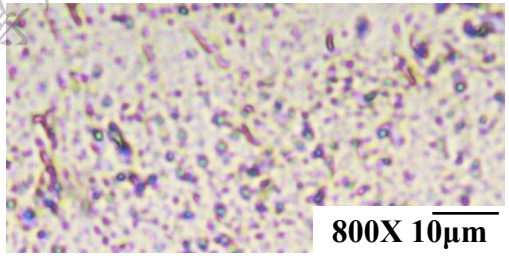

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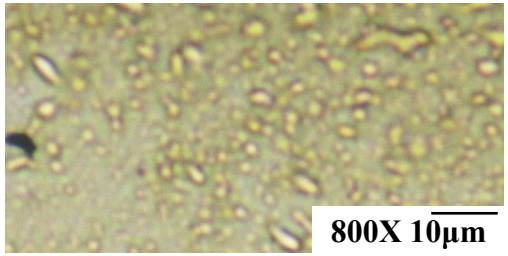

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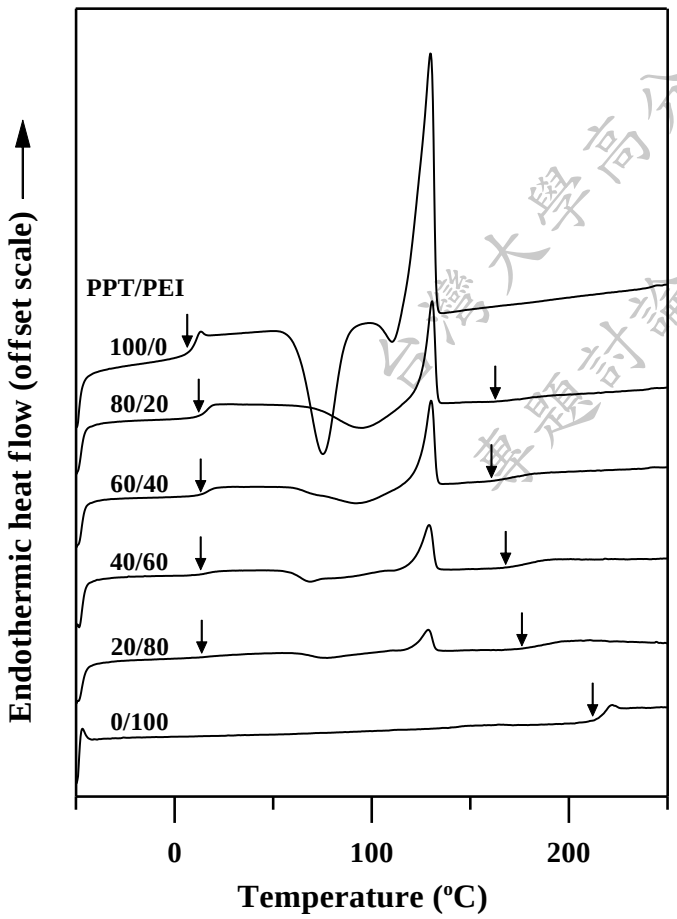
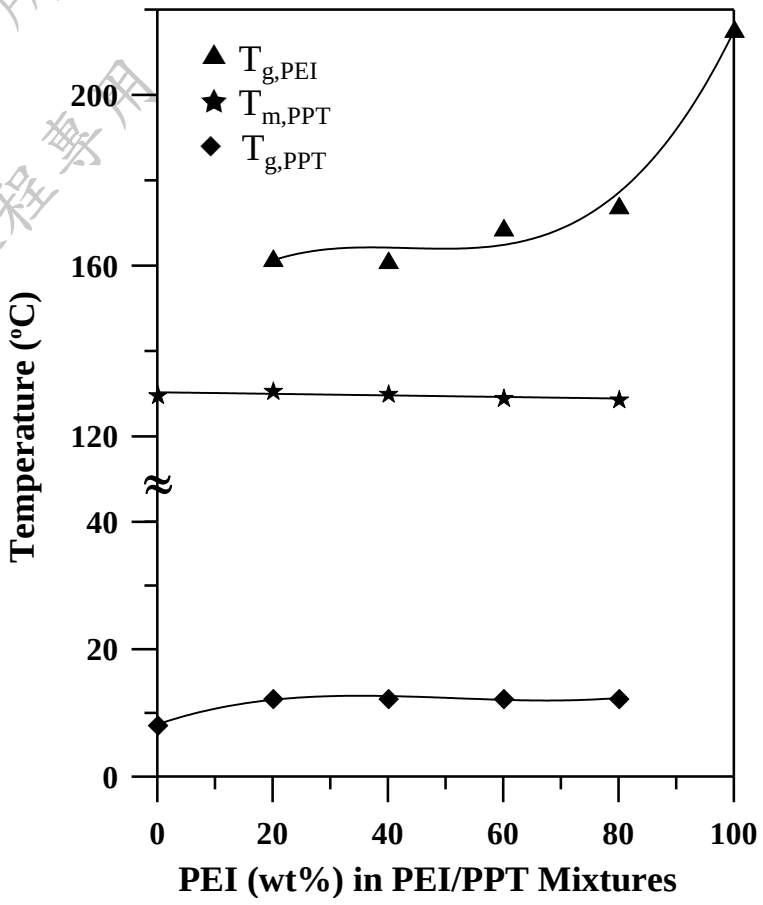
PPT/PEI binary blends

❖ **DSC traces of co-precipitated PPT/PEI blends**





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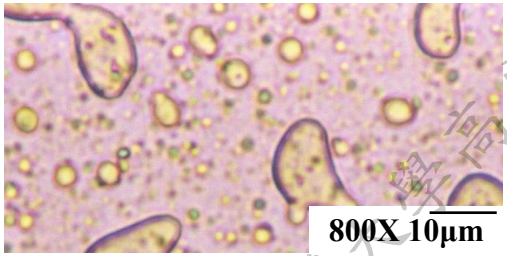
PHT/PEI binary blends

❖**POM graphs of co-precipitated PHT/PEI blends**



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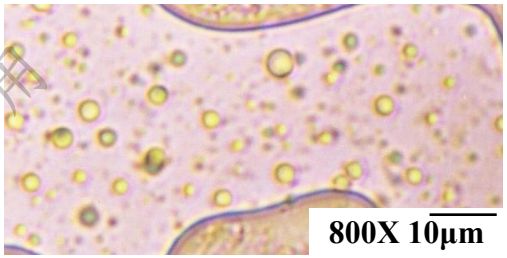

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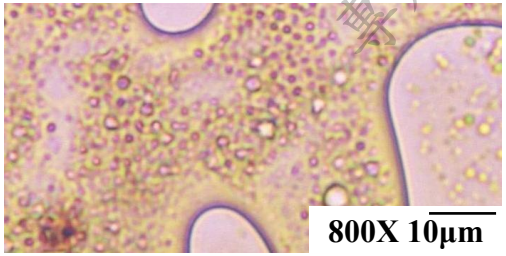

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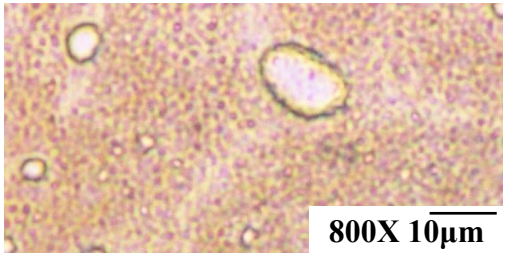

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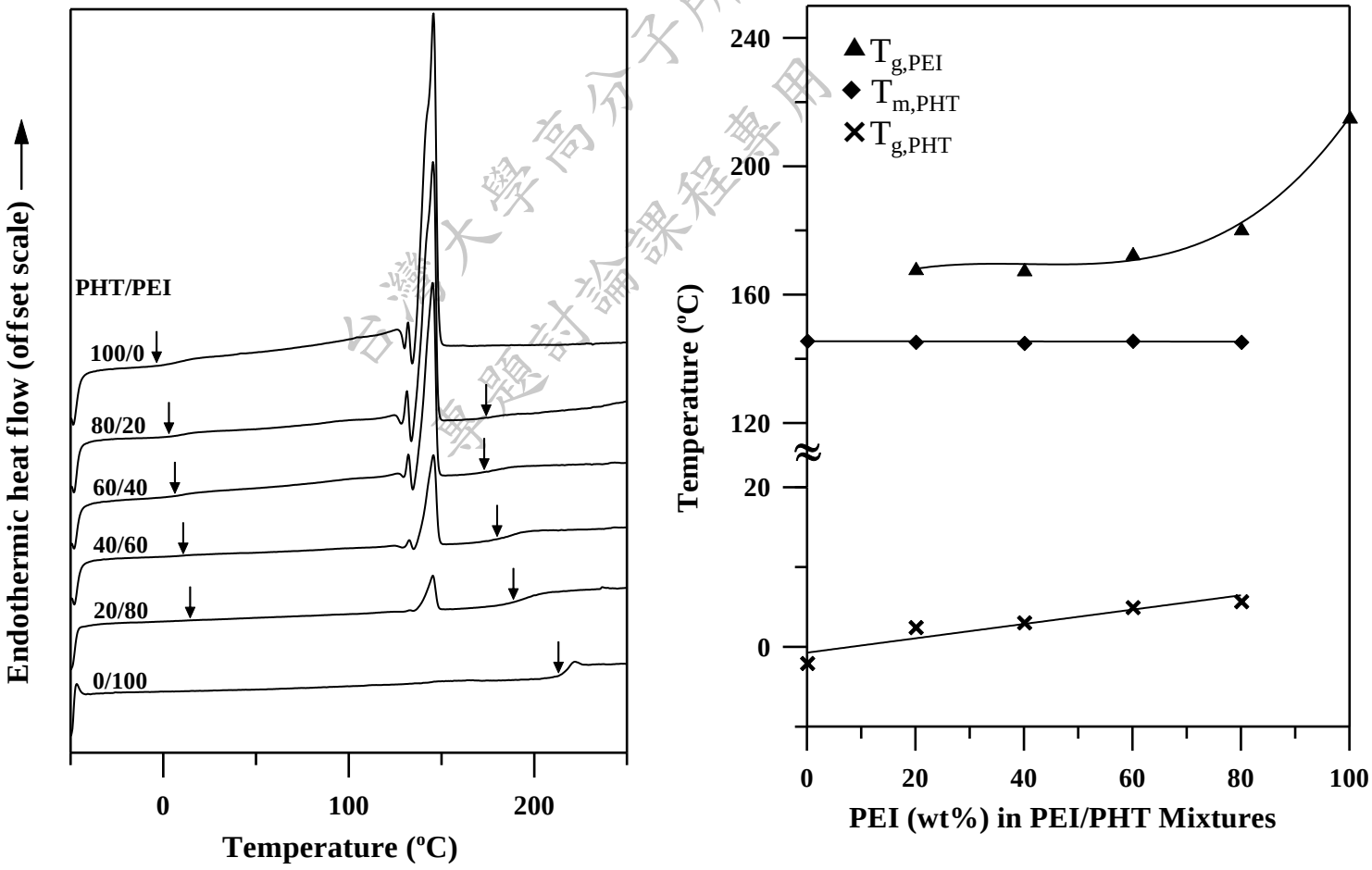
PHT/PEI binary blends

❖ **DSC traces of co-precipitated PHT/PEI blends**



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❖ PPT/PEI & PHT/PEI binary blends

❖ *Polymer-polymer interaction parameter*

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Polymer-polymer interaction parameter

❖ Modified Fox equation

$$\omega_{2,I} = 1 - \frac{T_{g1}(T_{g,I} - T_{g2})}{T_{g,I}(T_{g1} - T_{g2})}$$

❖ Modified Couchman equation

$$\omega_{2,I} = 1 - \frac{\Delta C_{p2}(\ln T_{g,I} - \ln T_{g2})}{\Delta C_{p1}(\ln T_{g1} - \ln T_{g,I}) + \Delta C_{p2}(\ln T_{g,I} - \ln T_{g2})}$$

$$m_1 = \frac{\overline{M}_{n,1}/\rho_1}{V_o} \qquad m_2 = \frac{\overline{M}_{n,2}/\rho_2}{V_o}$$

$$\chi_{12} = \frac{(\phi_{1,I}^{\ 2} - \phi_{1,II}^{\ 2})[m_2 \ln \frac{\phi_{1,II}}{\phi_{1,I}} + (m_1 - m_2)(\phi_{2,I} - \phi_{2,II})] + (\phi_{2,I}^{\ 2} - \phi_{2,II}^{\ 2})[m_1 \ln \frac{\phi_{2,II}}{\phi_{2,I}} + (m_2 - m_1)(\phi_{1,I} - \phi_{1,II})]}{2m_1 m_2 (\phi_{1,I}^{\ 2} - \phi_{1,II}^{\ 2})(\phi_{2,I}^{\ 2} - \phi_{2,II}^{\ 2})}$$

$$(\chi_{12})_c = \frac{1}{2}(m_1^{-\frac{1}{2}} + m_2^{-\frac{1}{2}})^2$$

χ_{12} : polymer-polymer interaction parameter

$\omega_{i,j}$: apparent weight fraction of polymer i in polymer j–rich phase

Ref : W. N. Kim and C. M. Burns, *Macromolecules*, **20,** 1876 (1987)

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PPT/PEI binary blends

❖ **Comparisons of χ_{12} calculated by Fox and Couchman equations**

χ_{12} of PPT/PEI blends				
	melt-blending		co-precipitation	
PEI wt%	$\chi_{12,Couchman}$	$\chi_{12,Fox}$	$\chi_{12,Couchman}$	$\chi_{12,Fox}$
10	0.129	0.129	--	--
20	0.119	0.120	0.125	0.125
30	0.118	0.118	--	--
40	0.116	0.116	0.125	0.125
50	0.115	0.115	--	--
60	0.113	0.113	0.127	0.127
70	0.113	0.113	--	--
80	0.113	0.113	0.129	0.130

⇨ We can derive the same values of χ_{12} by the two equations.

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PPT/PEI binary blends

- Calculated ω and χ_{12} of melt-blended and co-precipitated PPT/PEI blends by Fox equation.

PPT/PEI blends $(\chi_{12})_c$=0.086						
	melt-blending			co-precipitation		
	PPT-rich	PEI-rich		PPT-rich	PEI-rich	
PEI wt%	$\omega_{2,I}$	$\omega_{1,II}$	$\chi_{12,Fox}$	$\omega_{2,I}$	$\omega_{1,II}$	$\chi_{12,Fox}$
10	0.029	0.149	0.129	--	--	--
20	0.054	0.153	0.120	0.034	0.167	0.125
30	0.061	0.153	0.118	--	--	--
40	0.068	0.160	0.116	0.034	0.169	0.125
50	0.075	0.157	0.115	--	--	--
60	0.084	0.160	0.113	0.034	0.143	0.127
70	0.091	0.146	0.113	--	--	--
80	0.101	0.140	0.113	0.034	0.125	0.130

$\omega_{2,I}$：weight fraction of PEI in PPT-rich phase

$\omega_{1,II}$：weight fraction of PPT in PEI-rich phase

⇨ χ_{12} = 0.12±0.01 for PPT/PEI blends

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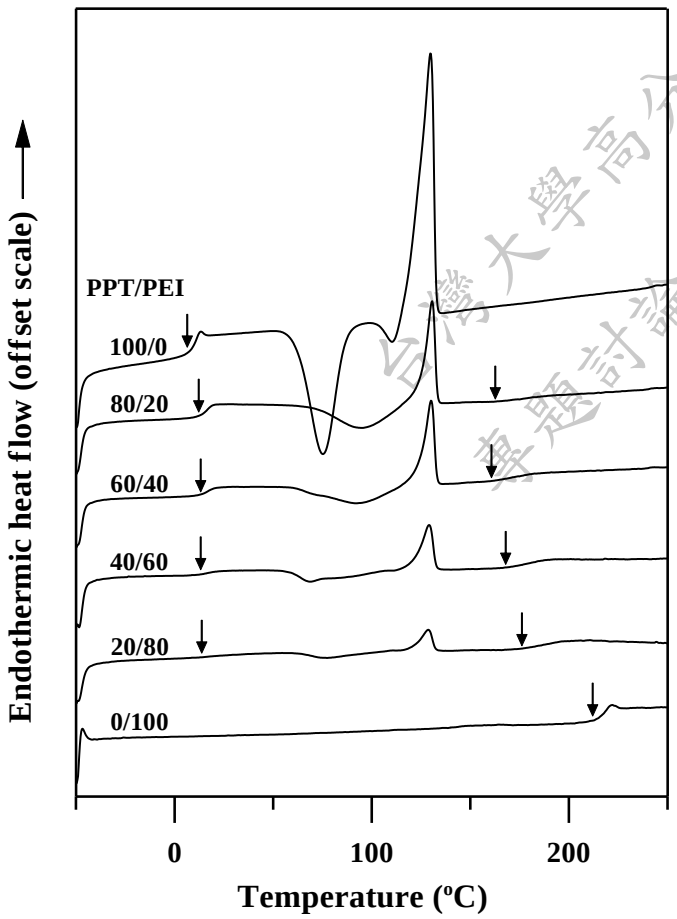
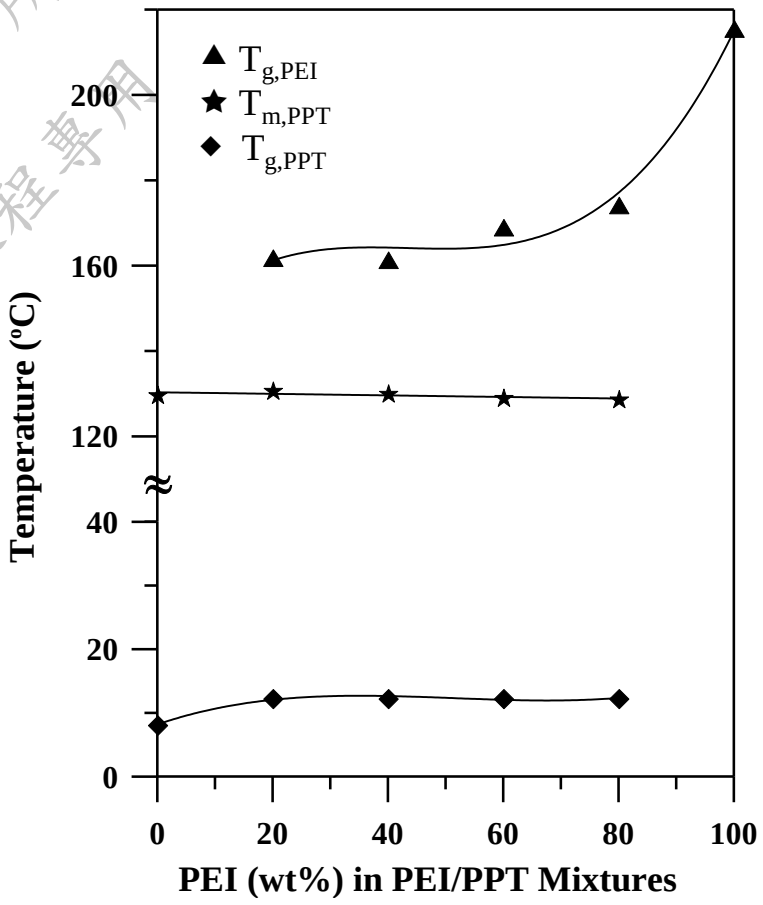
PPT/PEI binary blends

❖ **DSC traces of co-precipitated PPT/PEI blends**





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PHT/PEI binary blends

❖ Calculated ω and χ_{12} of co-precipitated PHT/PEI blends by Fox equation.

PHT/PEI blends $(\chi_{12})_c$=0.119					
	Fox equation				
	PHT-rich		PEI-rich		
PEI wt%	$\omega_{1,I}$	$\omega_{2,I}$	$\omega_{1,II}$	$\omega_{2,II}$	$\chi_{12,Fox}$
0	1.000	--	--	--	--
20	0.963	0.037	0.133	0.867	0.175
40	0.959	0.041	0.134	0.866	0.173
60	0.943	0.057	0.119	0.881	0.173
80	0.938	0.062	0.096	0.904	0.180
100	--	--	--	1.000	--

$\omega_{2,I}$：weight fraction of PEI in PHT-rich phase

$\omega_{1,II}$：weight fraction of PHT in PEI-rich phase

⇨ χ_{12} = 0.17±0.01 for PHT/PEI blends

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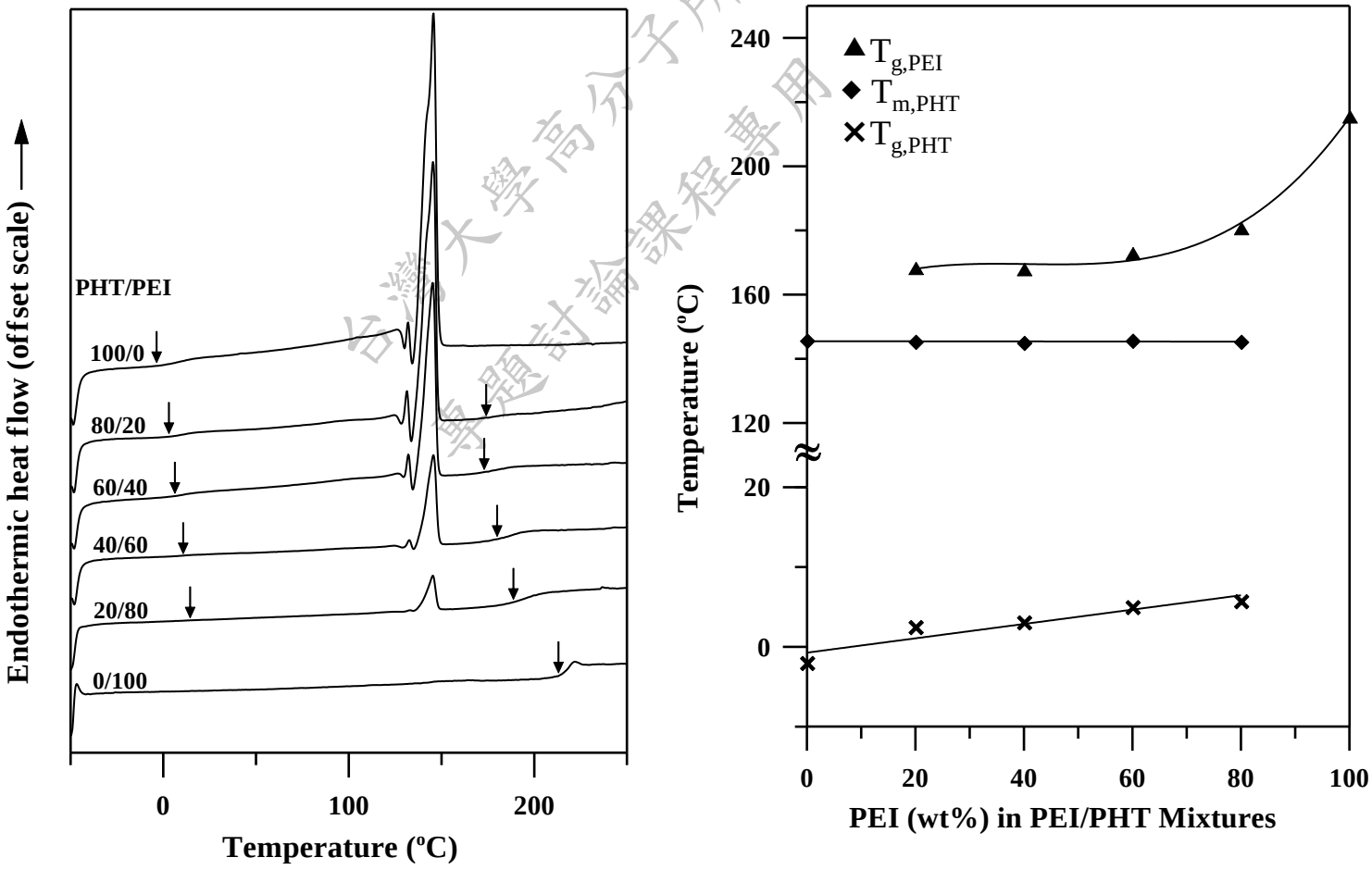
PHT/PEI binary blends

❖ **DSC traces of co-precipitated PHT/PEI blends**



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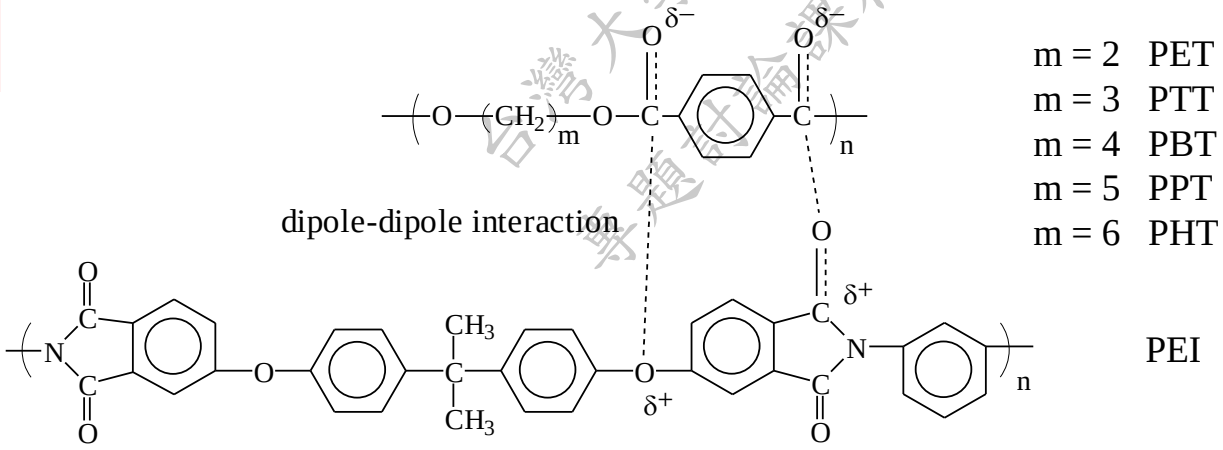
Polymer-polymer interaction parameter

Comparisons of aryl polyester/PEI blends

Aryl polyester/PEI	Method	T_g (^oC)	χ_{12}	Reference
PET/PEI	Melting-point depreesion	76	-0.66	Martin et al.[1]
PTT/PEI	Melting-point depreesion	42.8	-0.1	Wu and Woo[2]
PBT/PEI	Melting-point depreesion	32	-0.62	Yau and Woo[3]
PPT/PEI	Glass-transition temperature	8.1	0.12±0.01	This work
PHT/PEI	Glass-transition temperature	-2	0.17±0.01	This work



m = 2 PET
m = 3 PTT
m = 4 PBT
m = 5 PPT
m = 6 PHT

PEI

Ref : 1. J. M. Martin et al., *J. Appl. Polym. Sci.*, **48**, 935 (1993).

2. P. L. Wu, *Ph.D. thesis*, Department of Chemical Engineering, National Cheng Kung University, Tainan, Taiwan, 2003.

3. S. N. Yau and E. M. Woo, *Macromolecules*, **30**, 3626 (1997).

Part II Tenary blends

- **PEN/PPT/PEI & PEN/PTT/PEI ternary blends**

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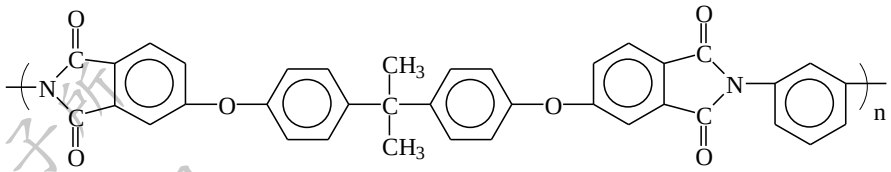
Materials

- **Poly(ether imide) PEI**
  Polysciences, Inc., USA
  M_w=30000 g mole^{-1} T_g=215.6℃



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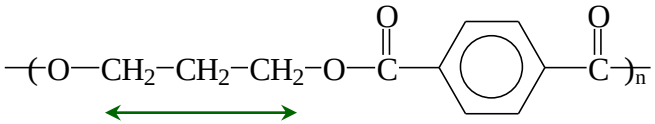
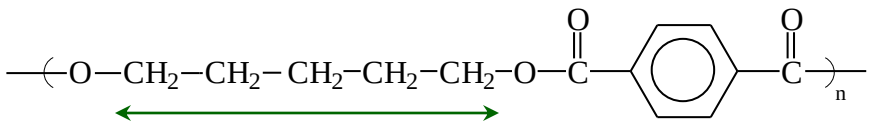
  ITRI.
  T_g=42.8℃，T_m=228.5℃



- **Poly(pentamethylene terephthalate) PPT**
  M_w=16600 g mole^{-1} PDI=1.54
  T_g=8.1℃，T_m=129.7℃



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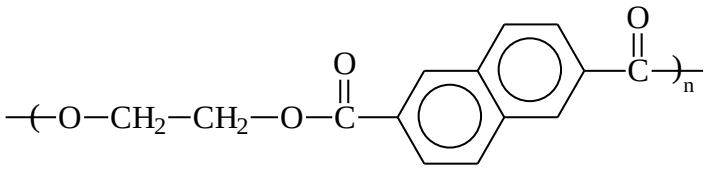
  Aldrich Inc.
  T_g =116 ℃，T_m=268℃



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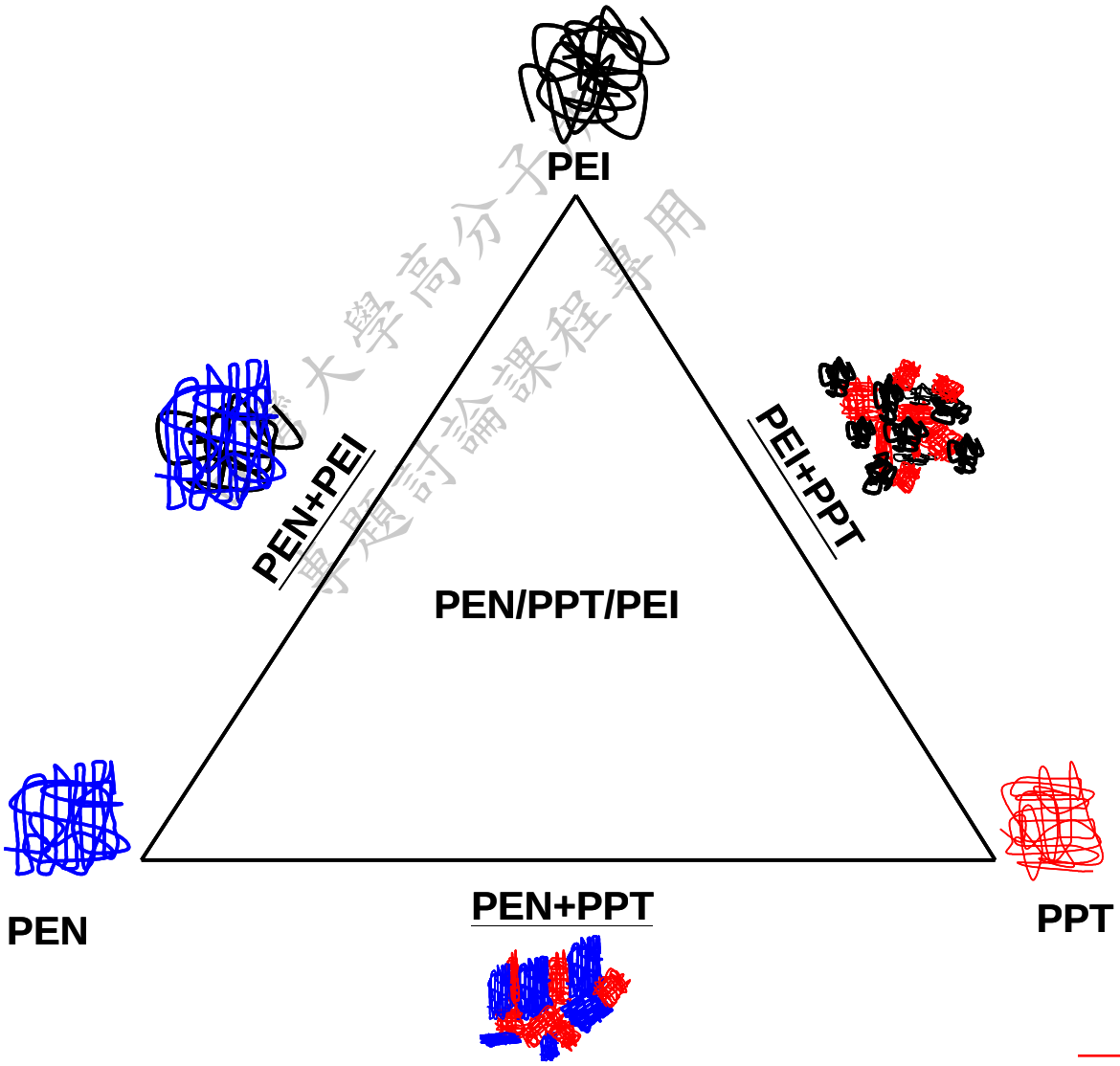
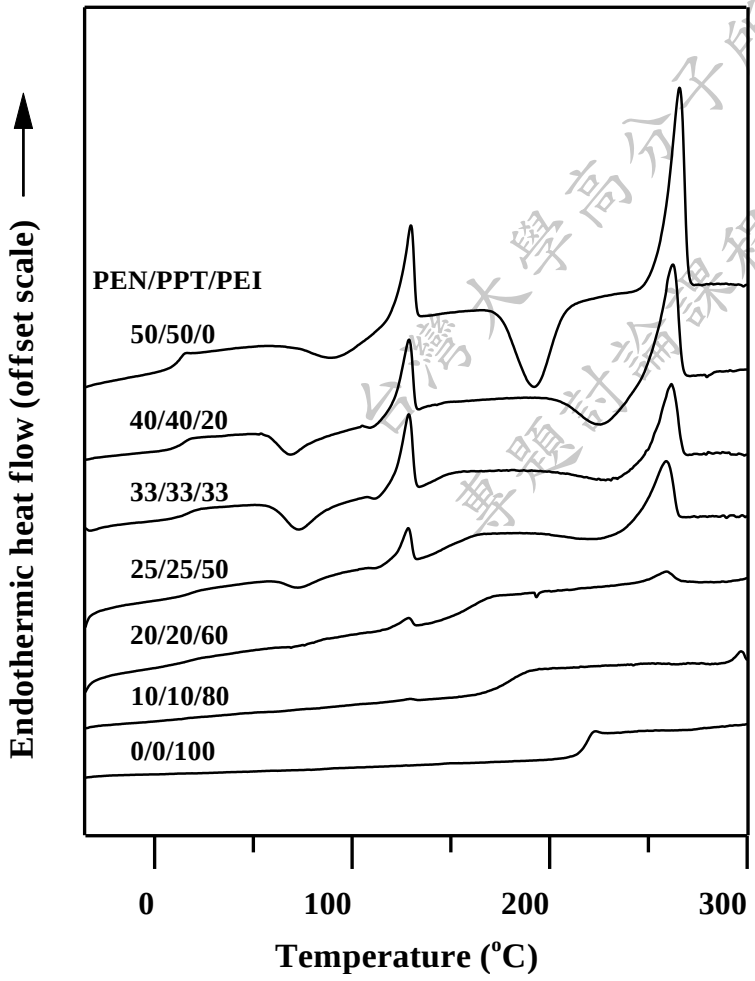
Miscibility of binary blends in a ternary polymer blend

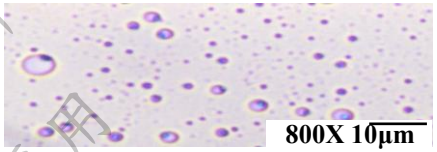


臺大學高分子專題討論課程專用

PEN/PPT/PEI ternary blends

❖ **DSC & POM results of PEN/PPT/PEI blends heated at 300°C for 1min**





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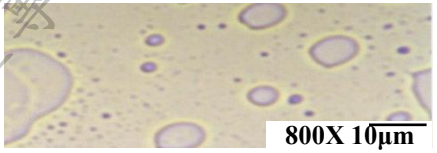

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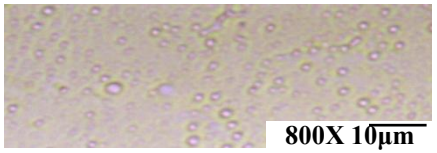
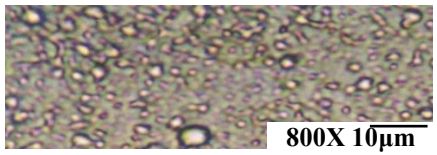
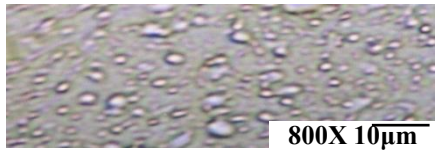


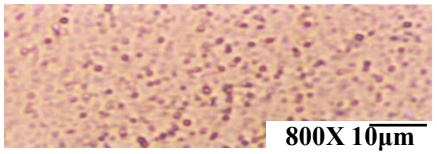
33/33/33



25/25/50



20/20/60



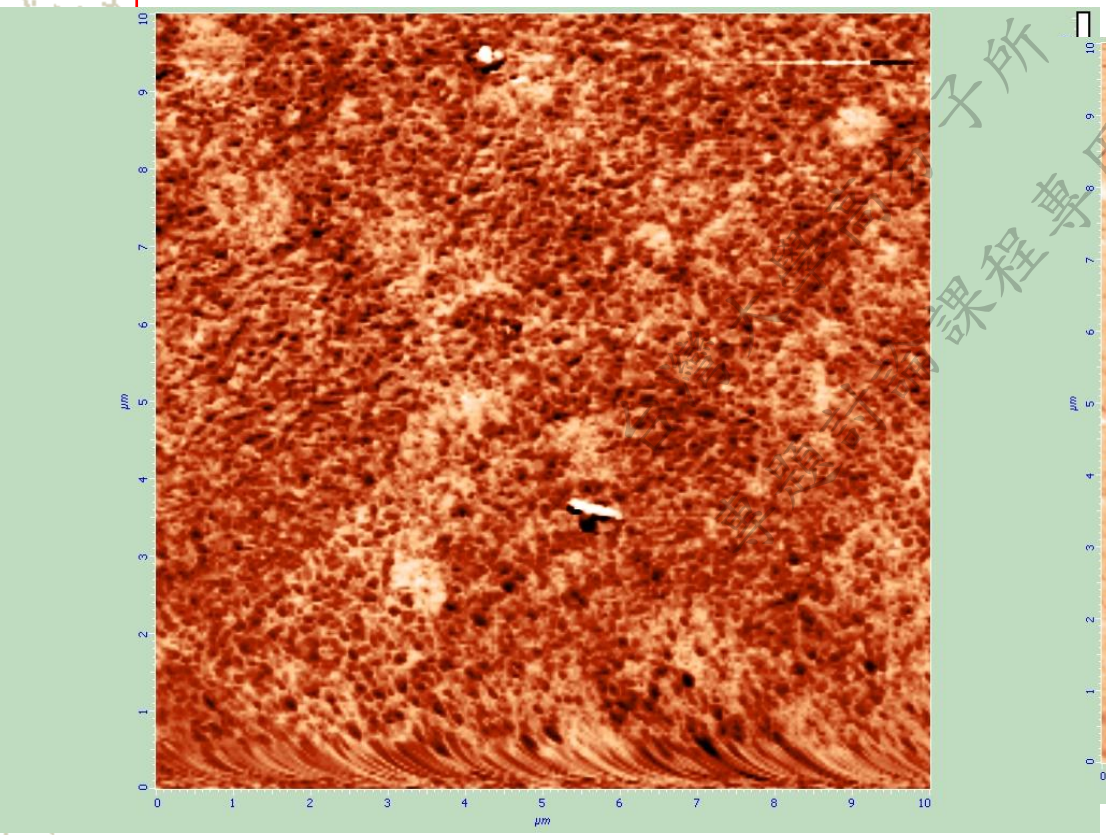
10/10/80

PEI wt%

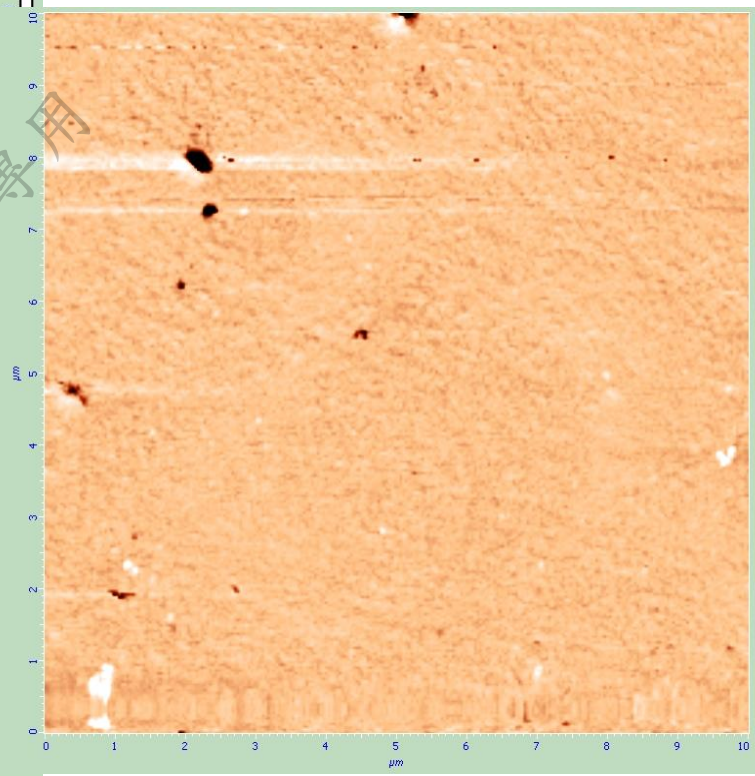
台灣大學高分子所 專題討論課程專用

PEN/PPT/PEI ternary blends

❖ **DSC & POM results of PEN/PPT/PEI =1/1/1 blends heated at 300°C**



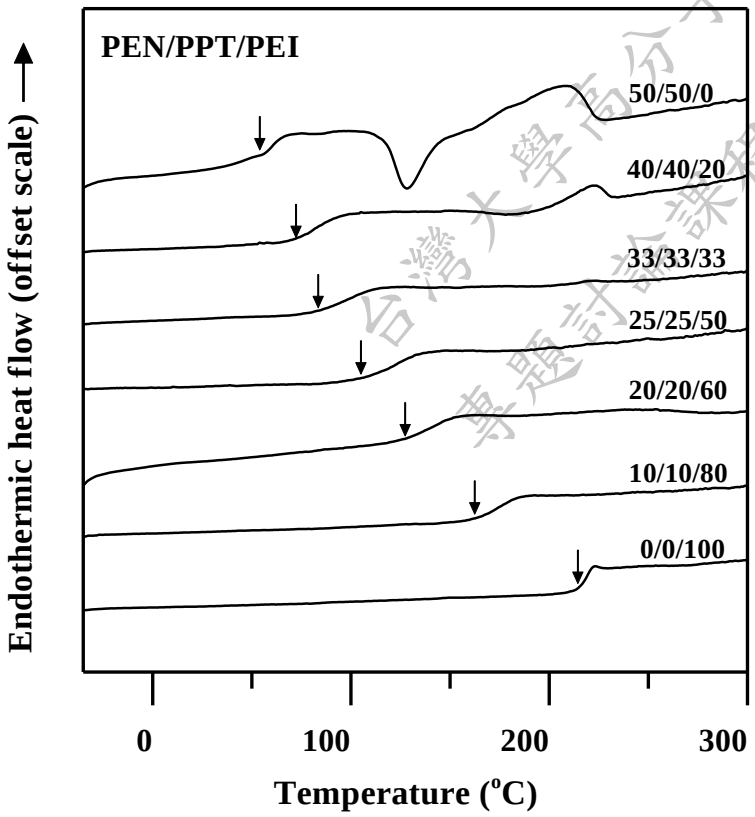
t= 1 min

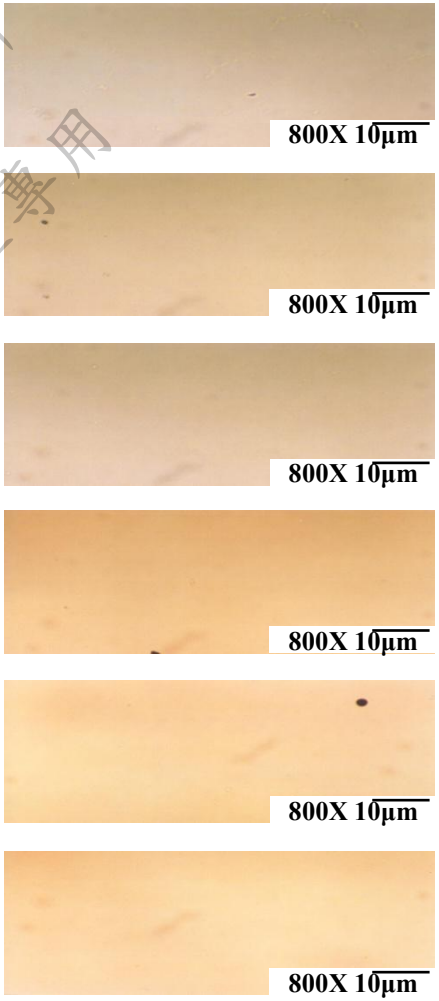


t= 60 min

PEN/PPT/PEI ternary blends

❖ **DSC & POM results of PEN/PPT/PEI blends heated at 300°C for 60min**

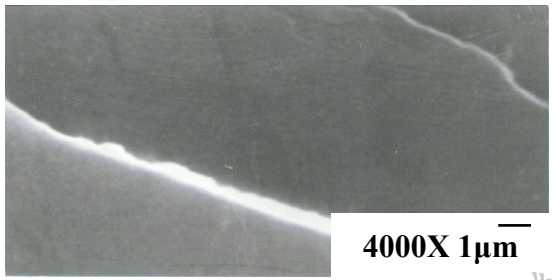




台灣大學高分子所
專題討論課程專用

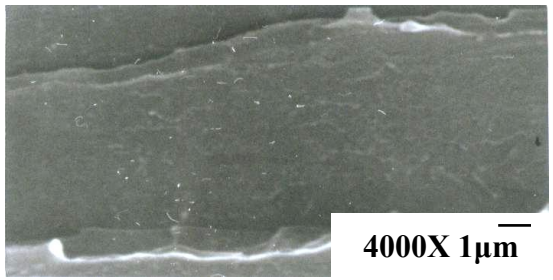
PEN/PPT/PEI ternary blends

❖SEM graphs of PEN/PPT/PEI blends heated at 300°C for 60min

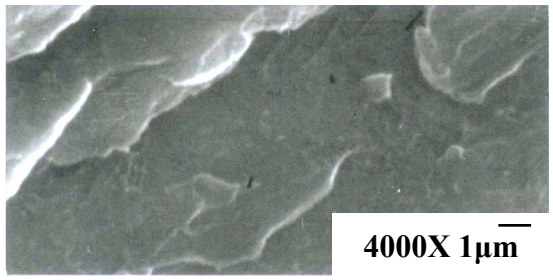


50/50/0

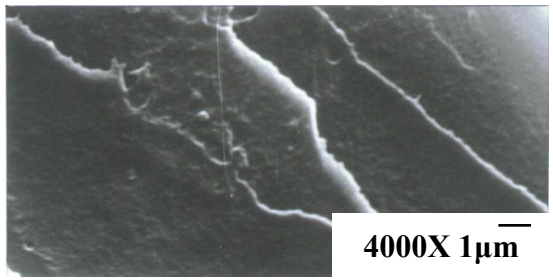
40/40/20



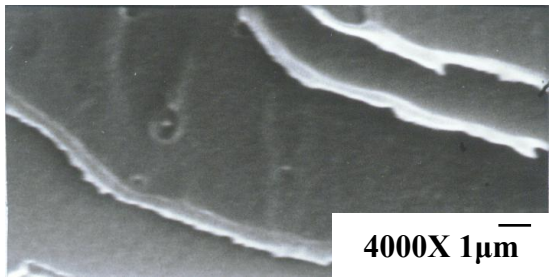
33/33/33



25/25/50



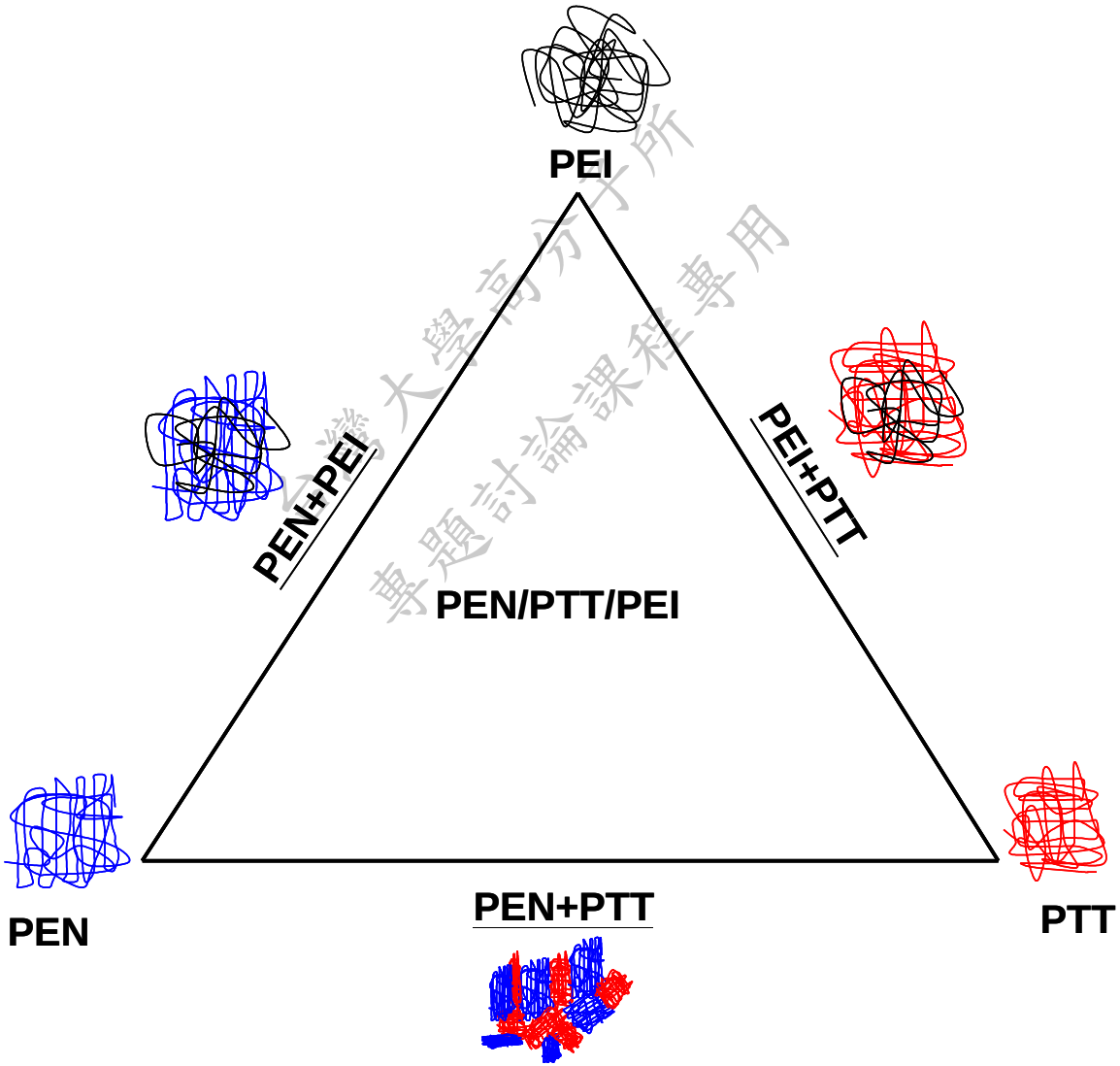
20/20/60



10/10/80

台灣大學化學所 專題討論課程

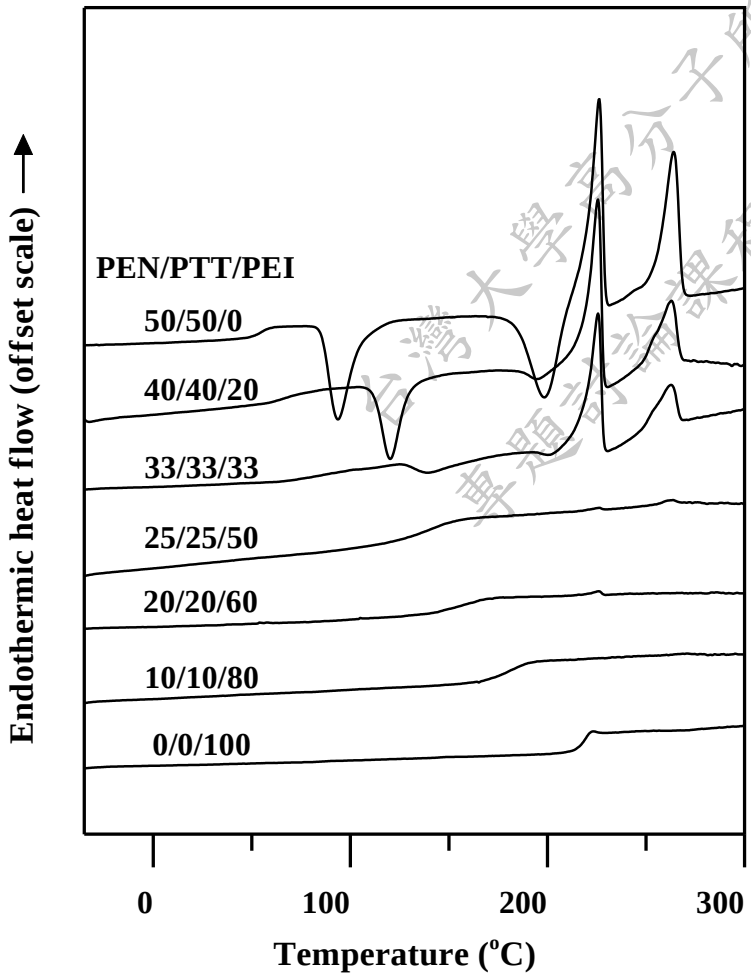
Miscibility of binary blends in a ternary polymer blend

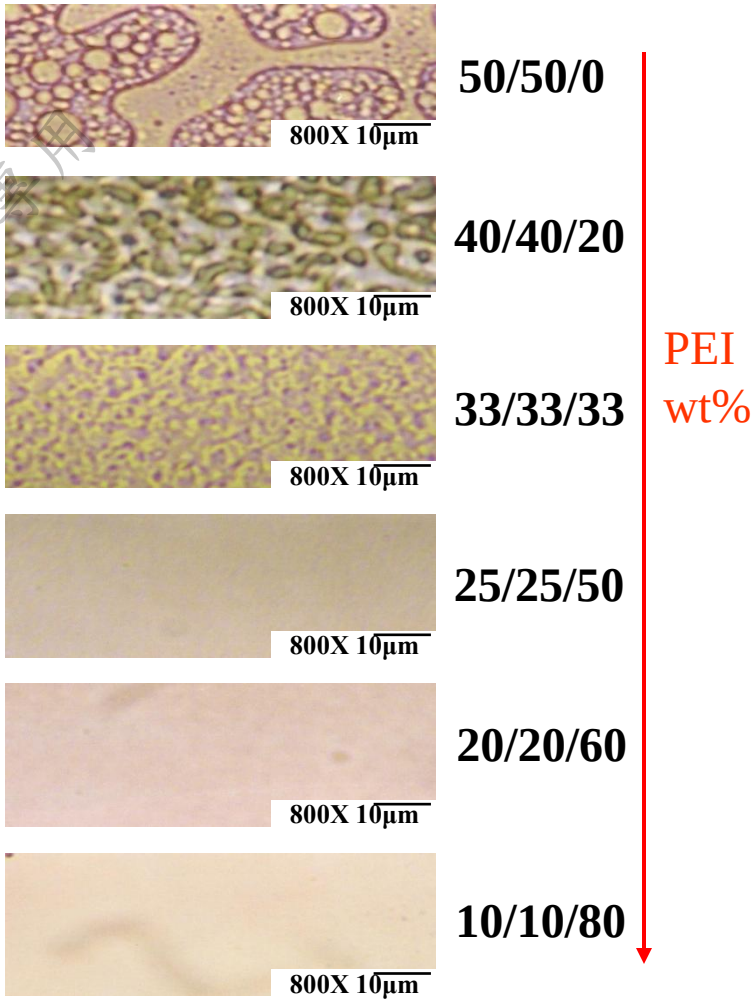


臺灣大學高分子所
專題討論課程專用

PEN/PTT/PEI ternary blends

❖ DSC & POM results of PEN/PTT/PEI blends heated at 300°C for 1min

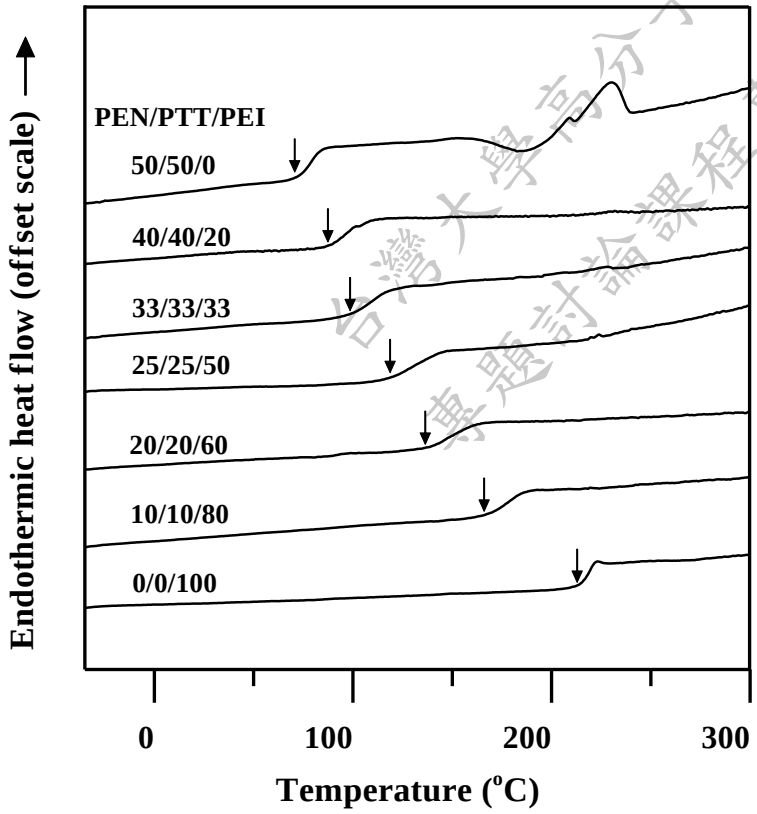




台灣大學高分子所 專題討論課程專用

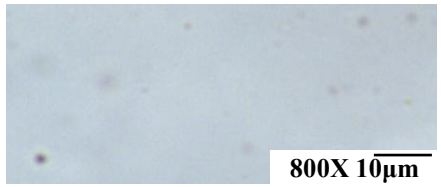
PEN/PTT/PEI ternary blends

❖ DSC & POM results of PEN/PTT/PEI blends heated at 300°C for 30min



50/50/0

40/40/20



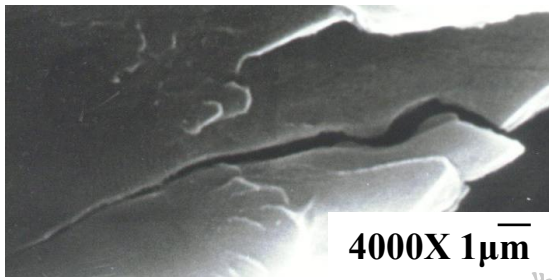
33/33/33

PEI wt%

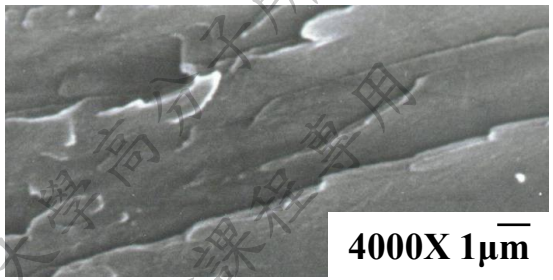
台灣大學高分子所 專題討論課程專用

PEN/PTT/PEI ternary blends

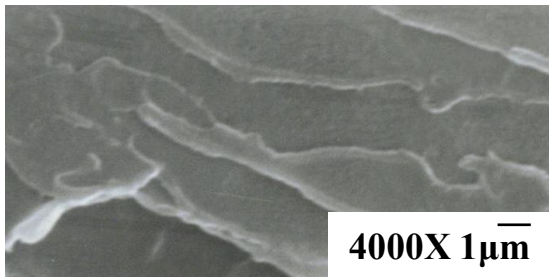
❖SEM graphs of PEN/PTT/PEI blends heated at 300°C 30min



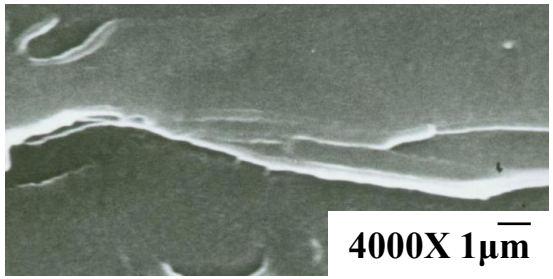
50/50/0



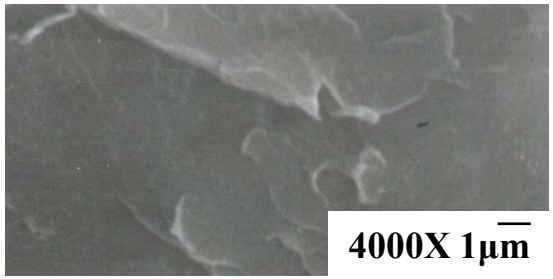
40/40/20



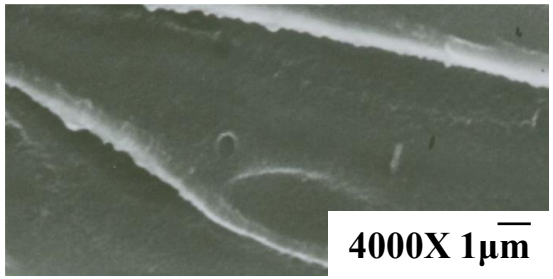
33/33/33



25/25/50



20/20/60



10/10/80

台灣大學高分子所 專題討論課程專用

PEN/PPT/PEI & PEN/PTT/PEI ternary blends

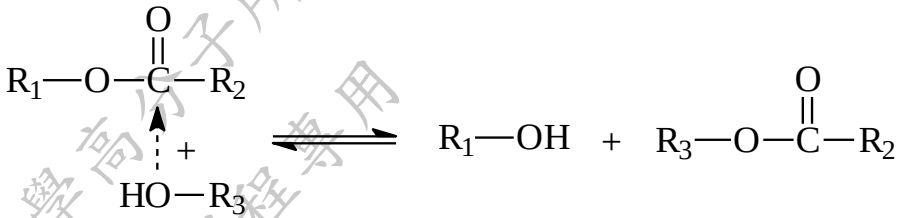
- *Transesterification reaction*

台灣大學高分子所
專題討論課程專用

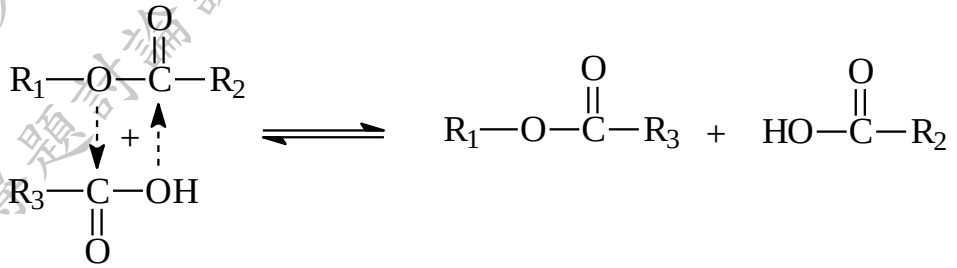
Transesterification reaction

❖ Mechanism

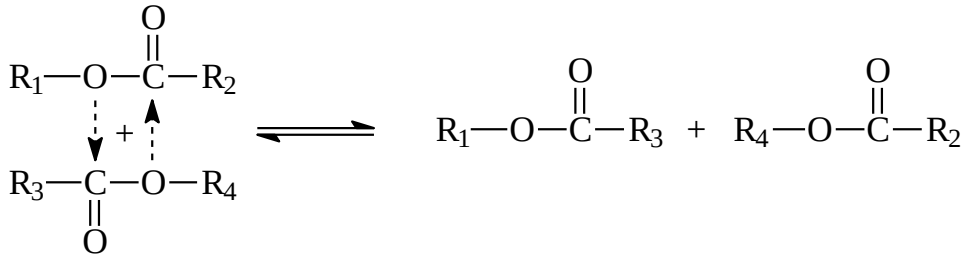
alcoholysis



acidolysis



direct ester-ester exchange (transesterification)

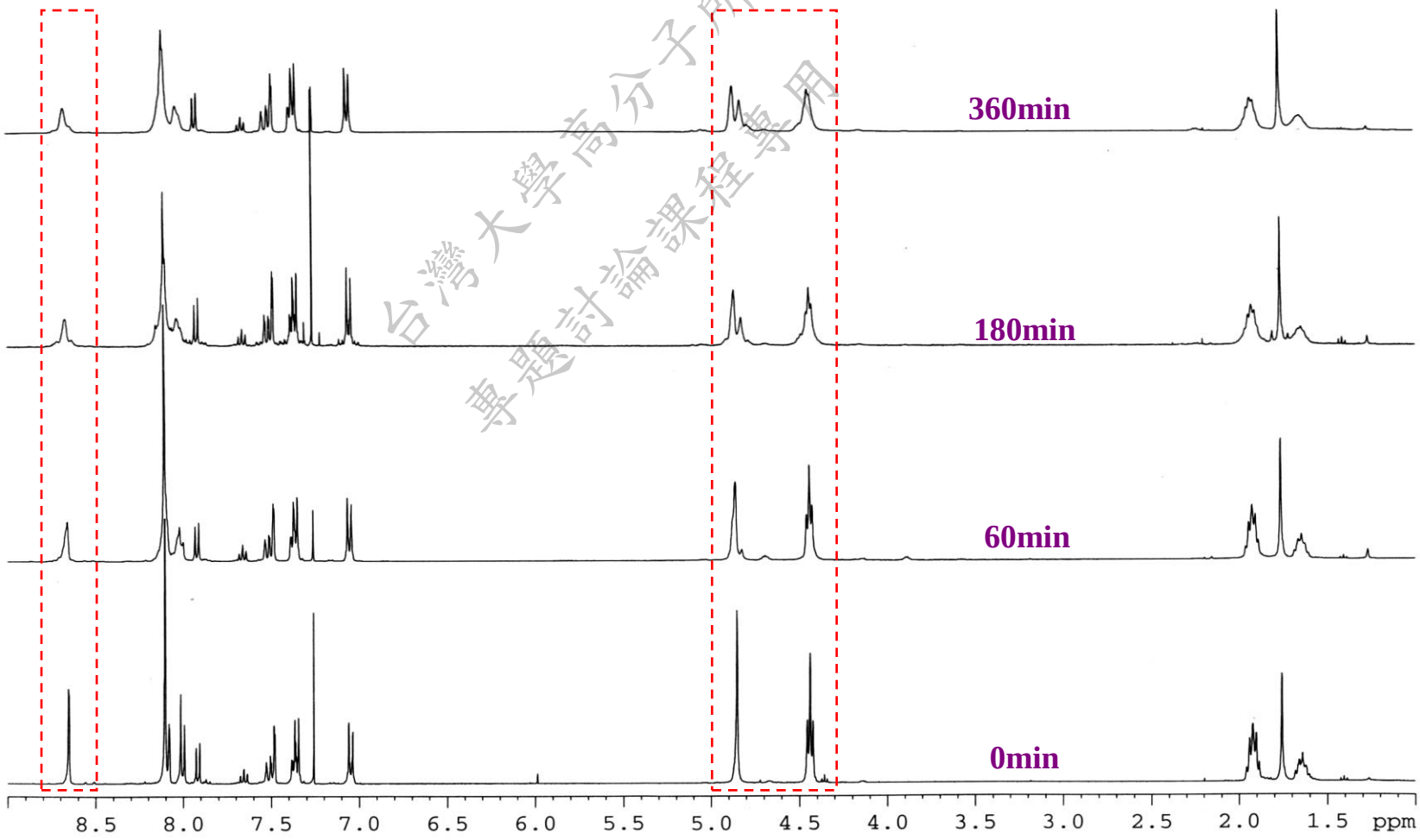


Ref : A. M. Kotliar, *J. Polym. Sci., Macromol. Rev.*, **16**, 367 (1981).

台灣大學高分子所 專題討論課程專用

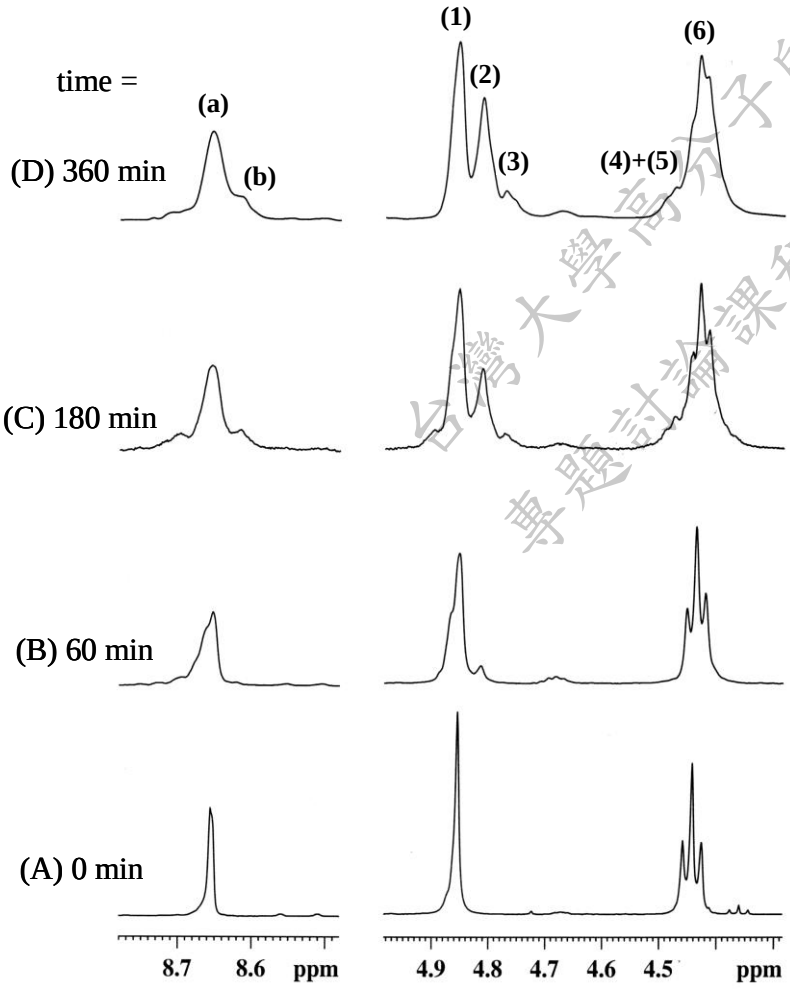
PEN/PPT/PEI ternary blends

❖ ^{1}H-NMR of PEN/PPT/PEI = 33/33/33 heated at 300°C for different times.

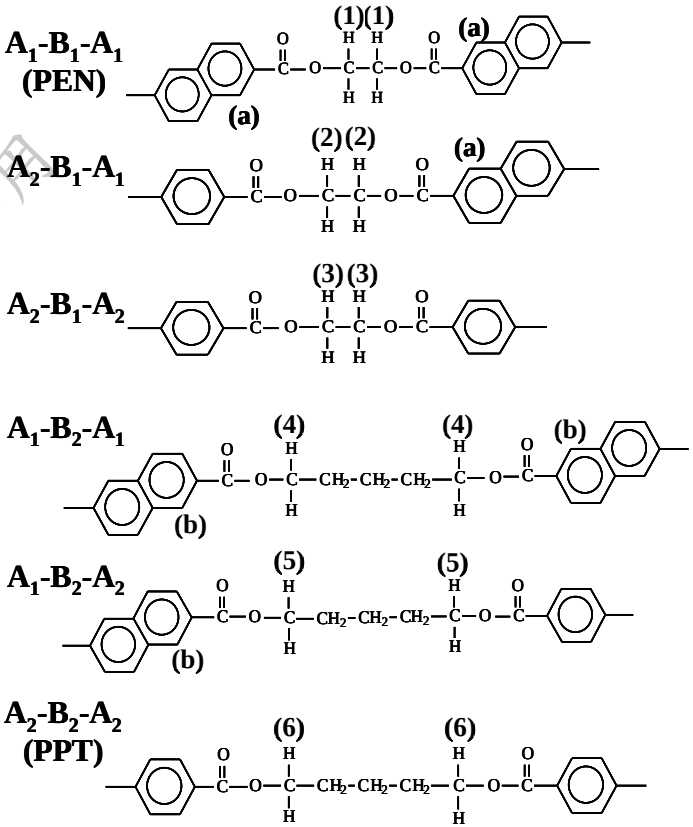


台灣大學高分子所
專題討論課程專用

PEN/PPT/PEI ternary blends



台灣大學高分子所 專題討論課程專用



A_1 : naphthalate B_1 : ethylene glycol

A_2 : terephthalate B_2 : pentamethylene glycol

Sequence distribution and degree of randomness

Molar fraction:

$$F_{A_iB_j} = [A_iB_j] \Big/ \sum_{i,j=1}^{2} [A_iB_j] = [A_iB_j] \Big/ \sum_{i=1}^{2} [A_i]$$

Probability:

$$P_{A_iB_j} = [A_iB_j] \Big/ \sum_{j=1}^{2} [A_iB_j] = \frac{[A_iB_j]}{[A_i]}$$

$$P_{A_iB_j} = \frac{F_{A_iB_j}}{F_{A_i}}$$

degree of randomness around B_j:

$$B = P_{A_iB_j} + P_{B_jA_i} = \frac{F_{A_iB_j}}{F_{A_i}} + \frac{F_{B_jA_i}}{F_{B_j}} \quad (i \neq j)$$

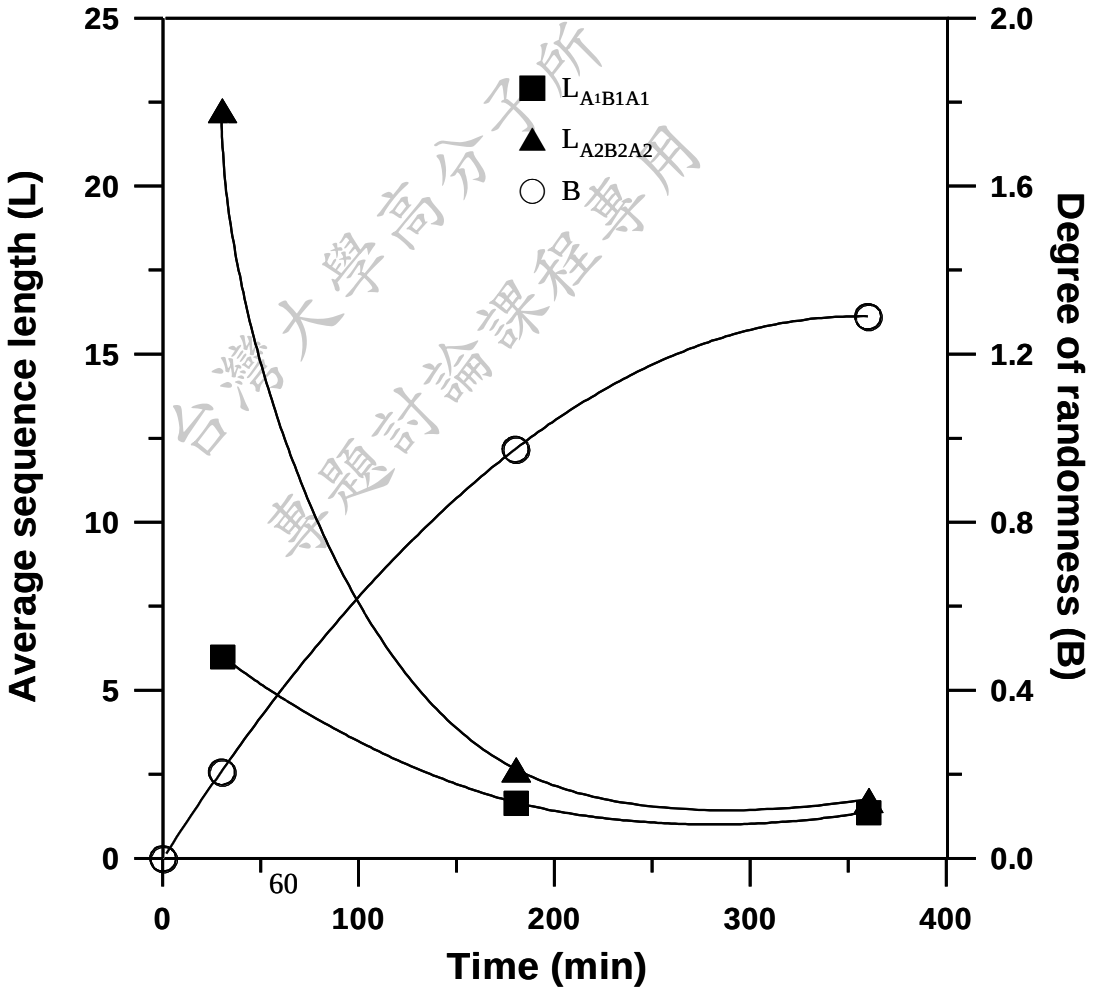
average sequence lengths:

$$x = L_{A_1B_1} = \frac{[A_1B_1]}{[A_1B_1A_2]} = \frac{1}{P_{A_1B_2A_2}}$$

台灣大學高分子所 專題討論課程專用

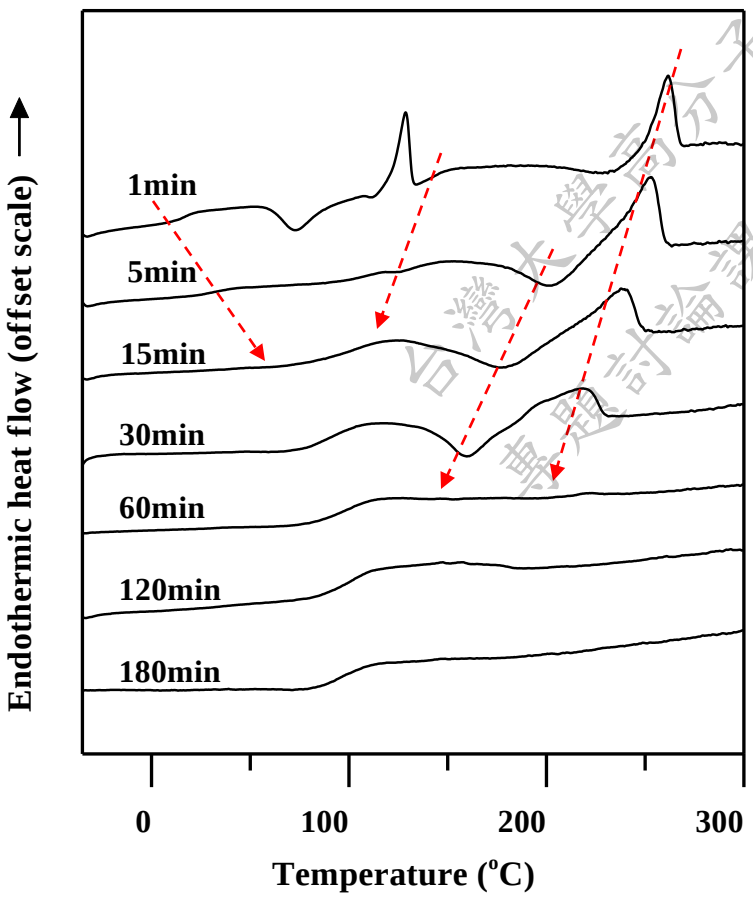
*Devaux J, Godard P, Mercier JP (1982) J Polym Sci Part B Polym Phys Ed 20:1875

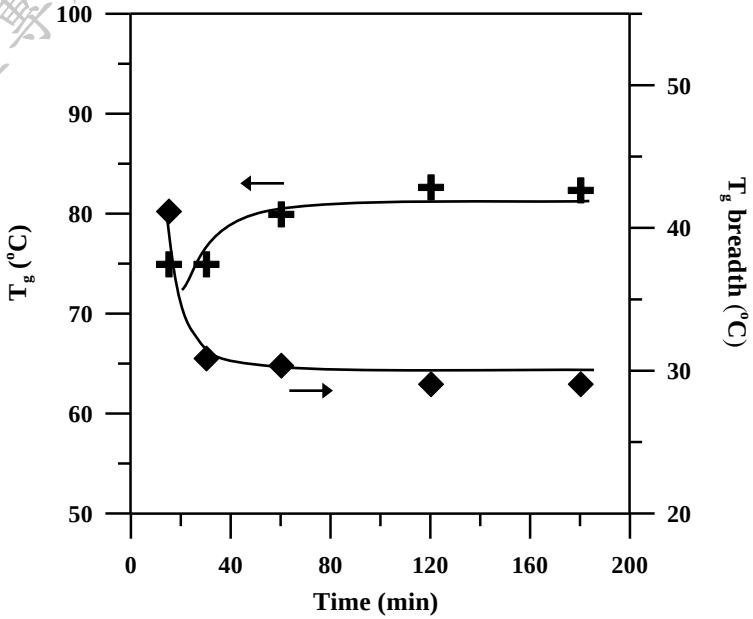
The average sequence length for PEN and PTT repeat units and the degree of randomness in the copolymer mixtures



PEN/PPT/PEI ternary blends

❖DSC traces of PEN/PPT/PEI=33/33/33 heated at 300°C for different times.



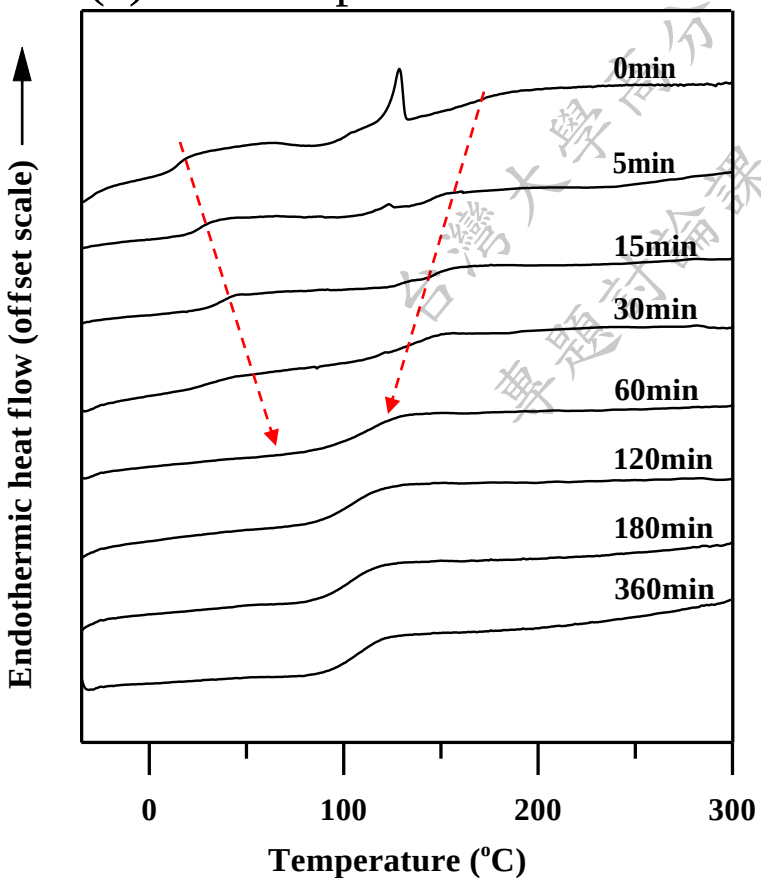


臺灣大學高分子所
專題討論課程專用

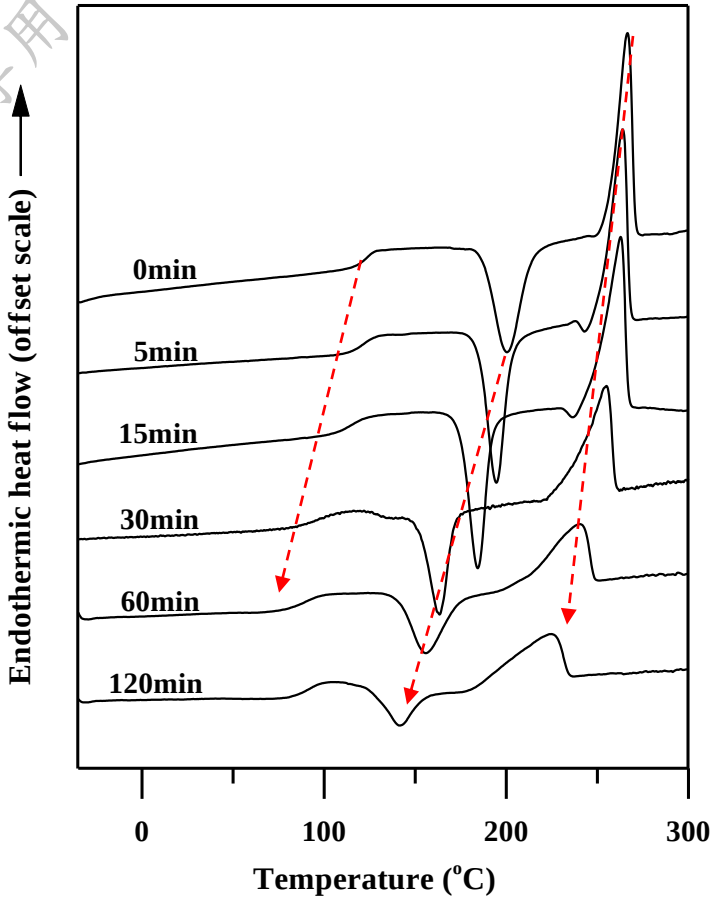
PEN/PPT/PEI ternary blends

- **Extraction experiment of PEN/PPT/PEI=33/33/33 heated at 300°C for different times. (solvent : chloroform)**

(a) Soluble portion



(b) Insoluble portion

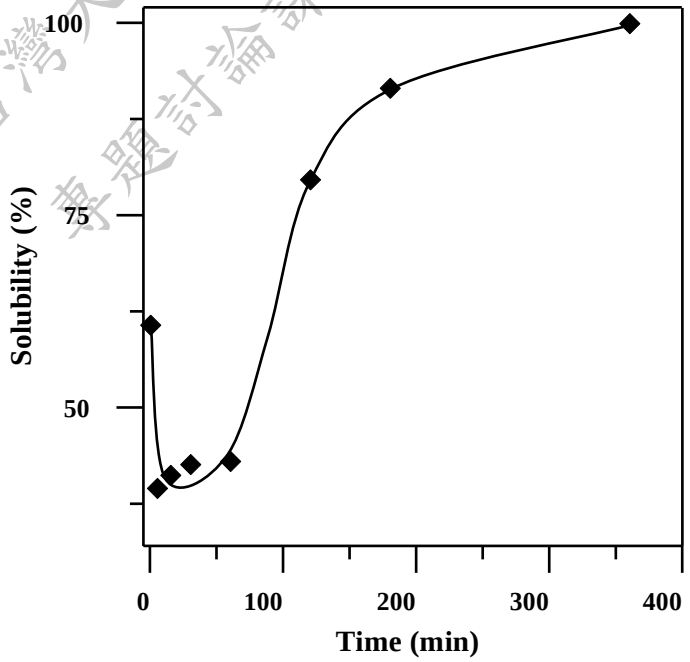


台灣大學高分子所 專題討論課程專用

PEN/PPT/PEI ternary Blends

- Solubility of PEN/PPT/PEI = 33/33/33 heated at 300°C for different times. (solvent : chloroform)

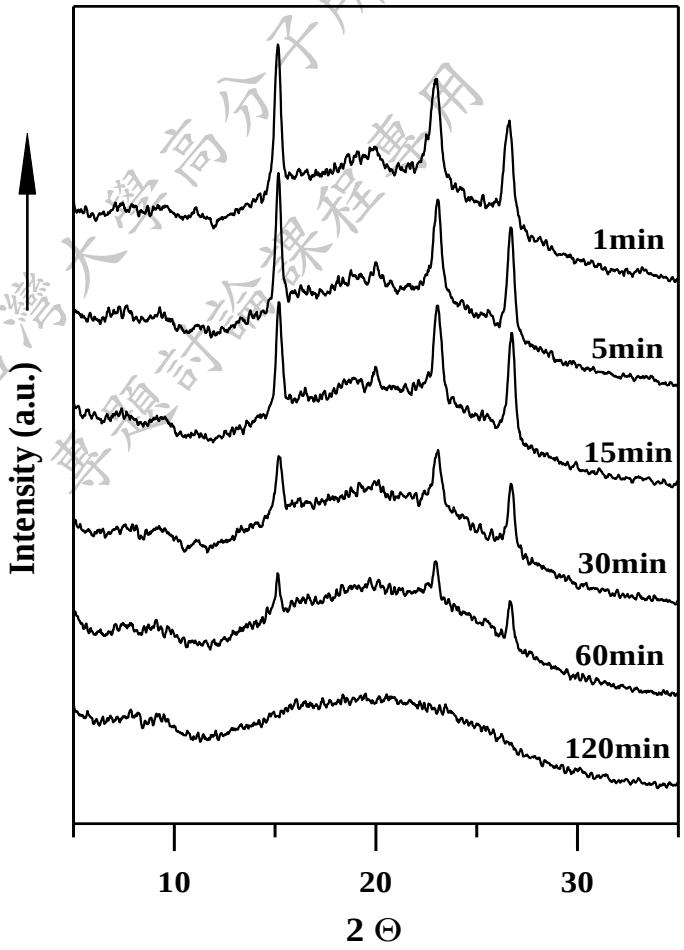
Time (min)	0	5	15	30	60	120	180	360
Solubility (%)	60.8	39.6	41.3	42.7	43.1	79.7	91.6	100



台灣大學高分子所 專題討論課程專用

PEN/PPT/PEI ternary blends

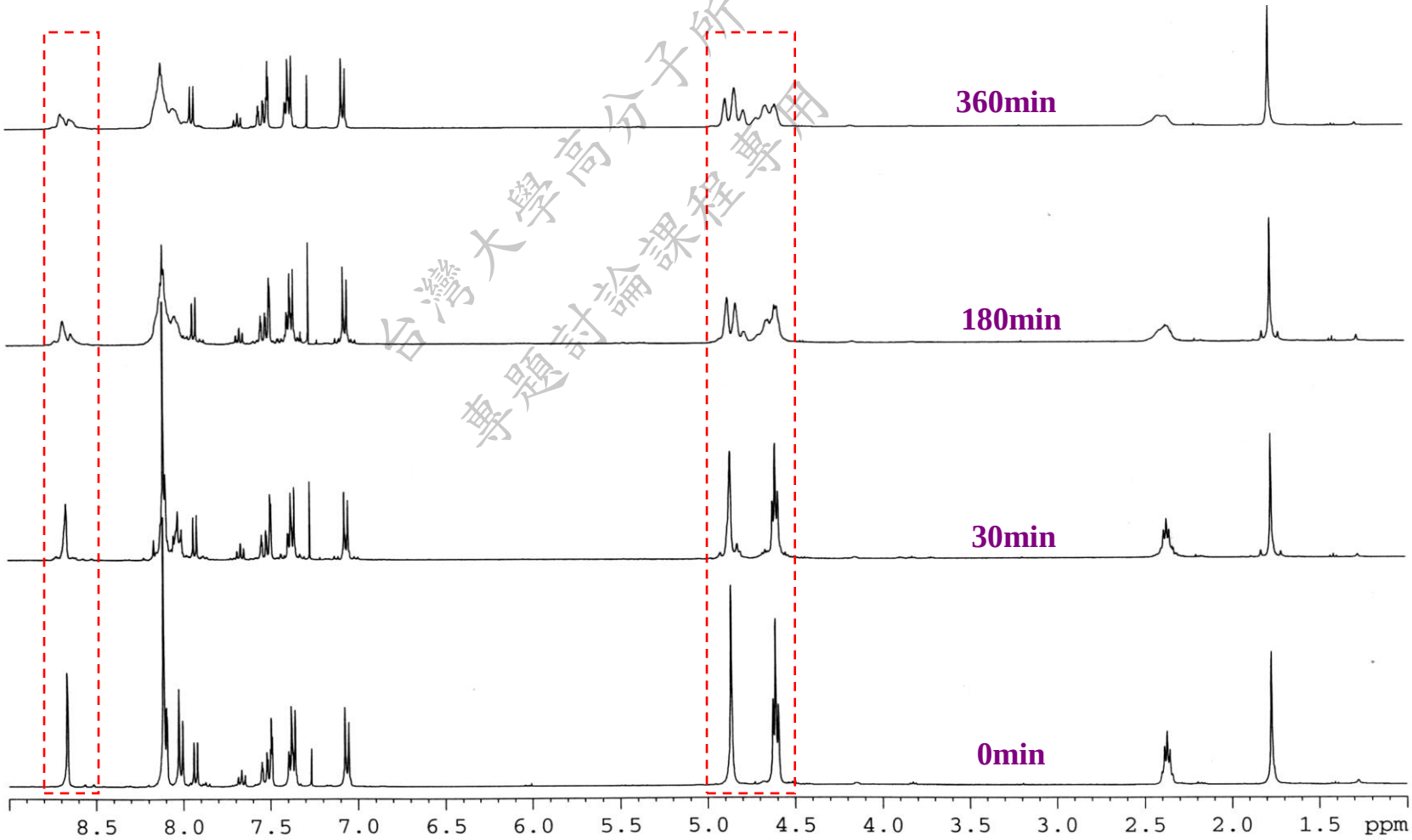
- XRD pattern of PEN/PPT/PEI=33/33/33 heated at 300°C for different times. (melt-crystallization at 200°C for 8hr)



台灣大學高分子所 專題討論課程專用

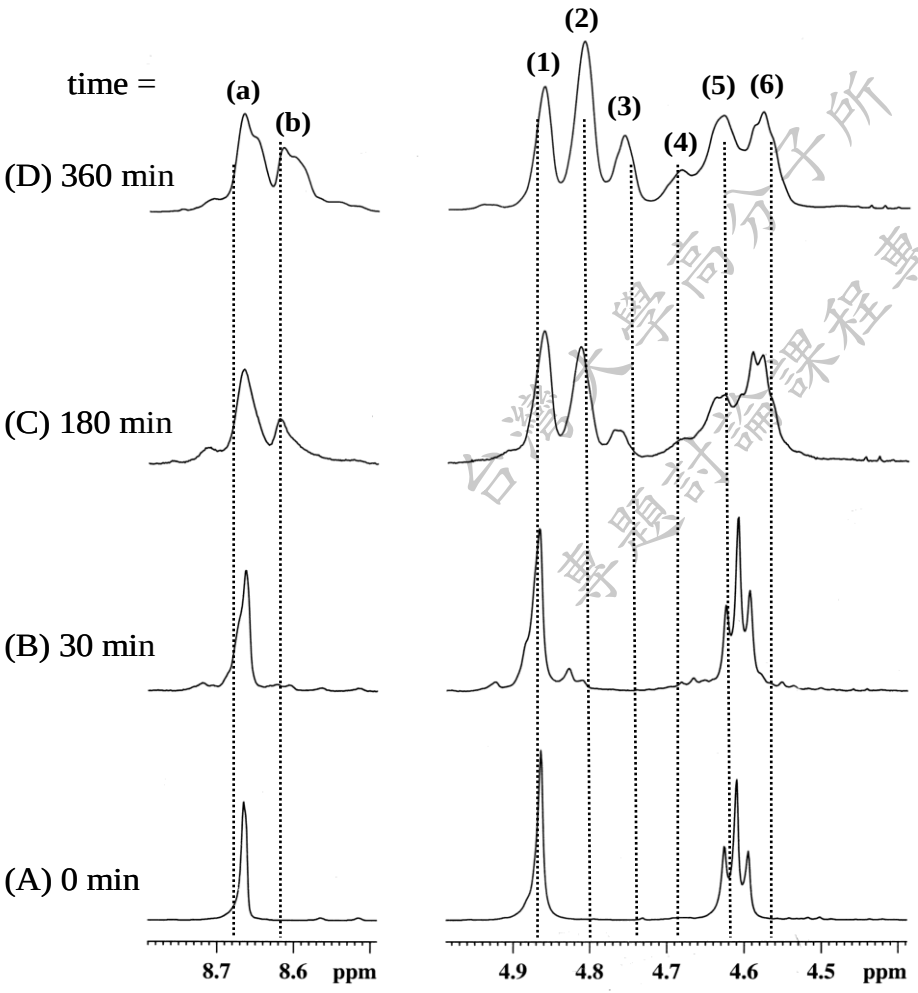
PEN/PTT/PEI ternary blends

❖ ^{1}H-NMR of PEN/PTT/PEI = 33/33/33 heated at 300^{o}C for different times.



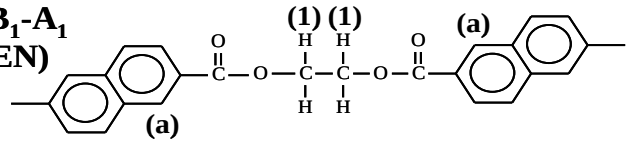
台灣大學高分子所
專題討論課程專用

PEN/PTT/PEI ternary blends

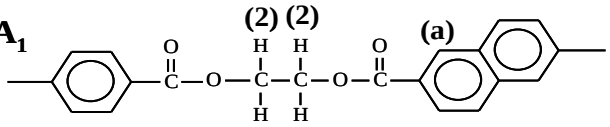


台灣大學高分子所 專題討論課程專用

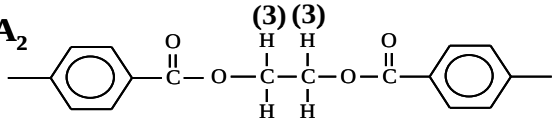
A_1-B_1-A_1 (PEN)



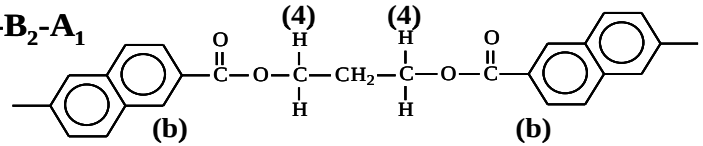
A_2-B_1-A_1



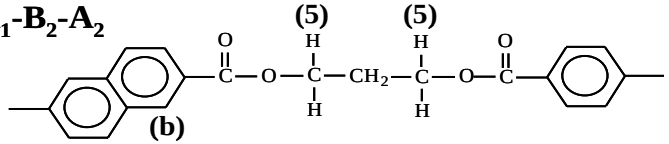
A_2-B_1-A_2



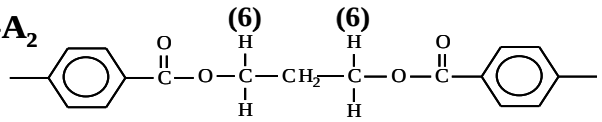
A_1-B_2-A_1



A_1-B_2-A_2



A_2-B_2-A_2 (PTT)

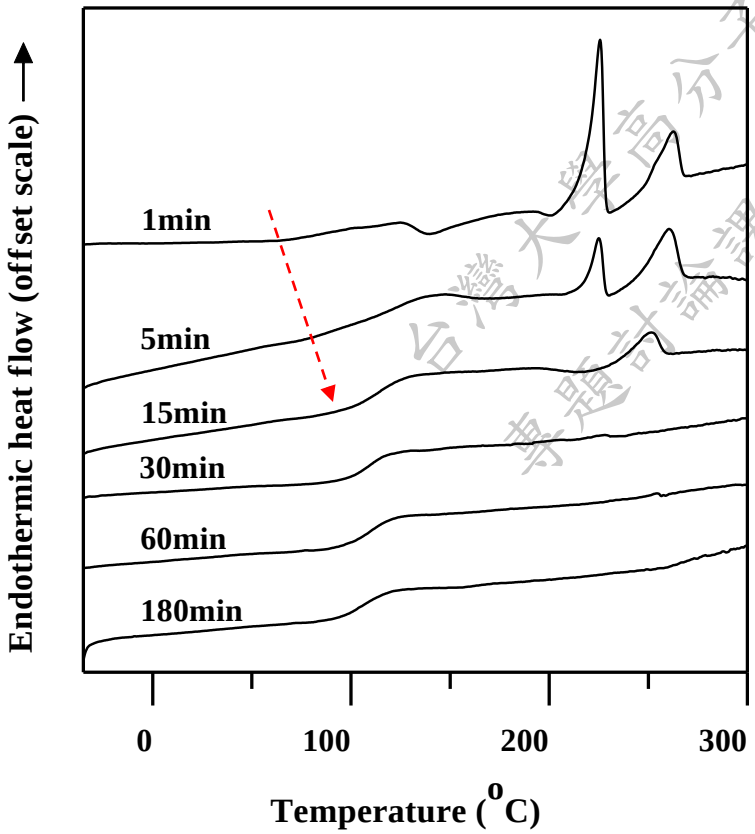


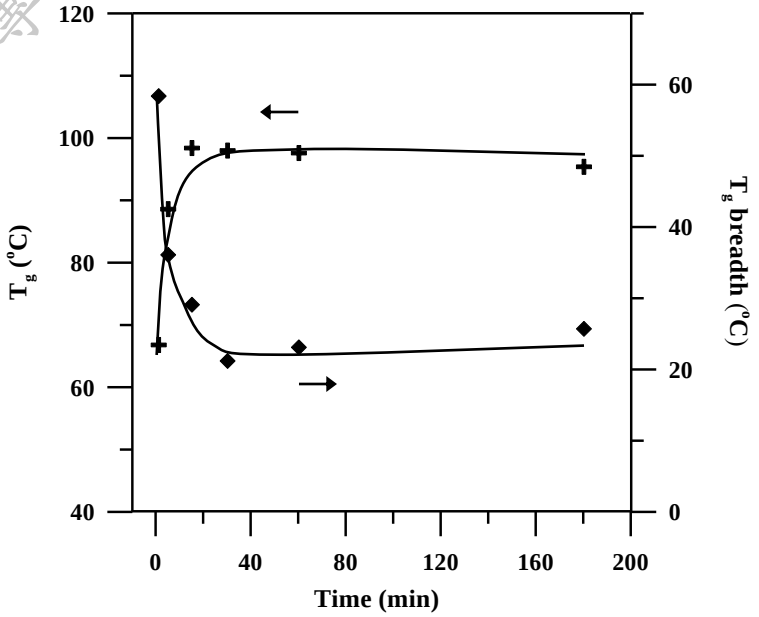
A_1 : naphthalate B_1 : ethylene glycol

A_2 : terephthalate B_2 : trimethylene glycol

PEN/PTT/PEI ternary blends

❖DSC traces of PEN/PTT/PEI=33/33/33 heated at 300°C for different times.

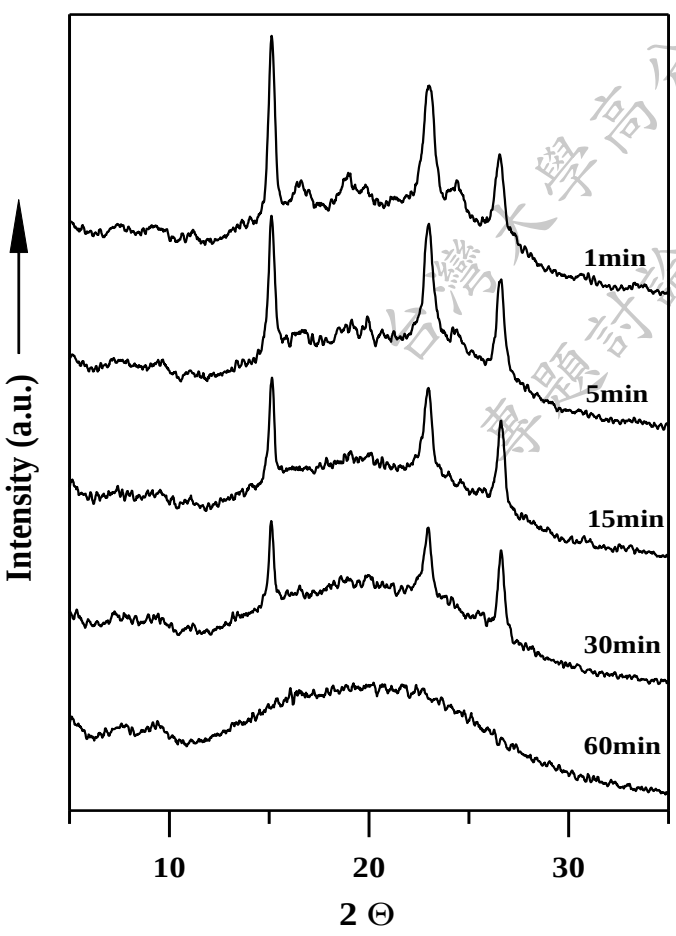


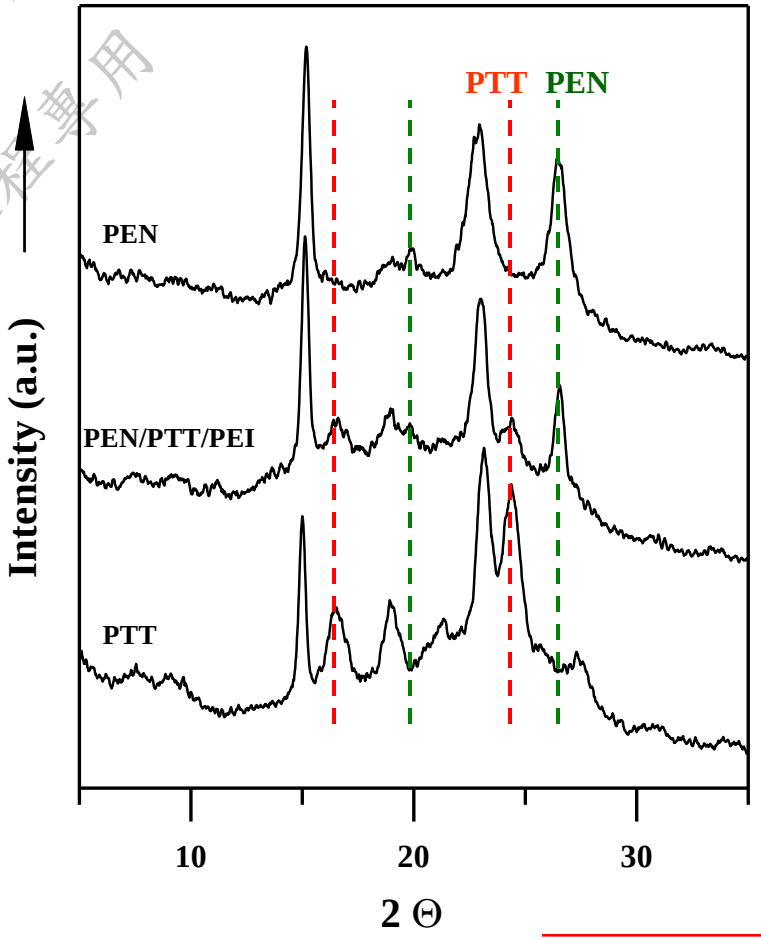


台灣大學高分子所 專題討論課程專用

PEN/PTT/PEI ternary blends

- XRD pattern of PEN/PTT/PEI=33/33/33 heated at 300°C for different times. (melt-crystallization at 200°C for 8hr)





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Compatibilizers --- ENPT and ENTT copolymers

Compatibility

- ENPT/PEI binary blends
- ENTT/PEI binary blends
- ENPT/PPT/PEI ternary blends

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❖ What are the ENPT and ENTT copolymers?

ENPT : the copolymer obtained by melt-blending PEN/PPT=5/5 (w/w) at 300°C for 6hr.

ENTT : the copolymer obtained by melt-blending PEN/PTT=5/5 (w/w) at 300°C for 6hr.

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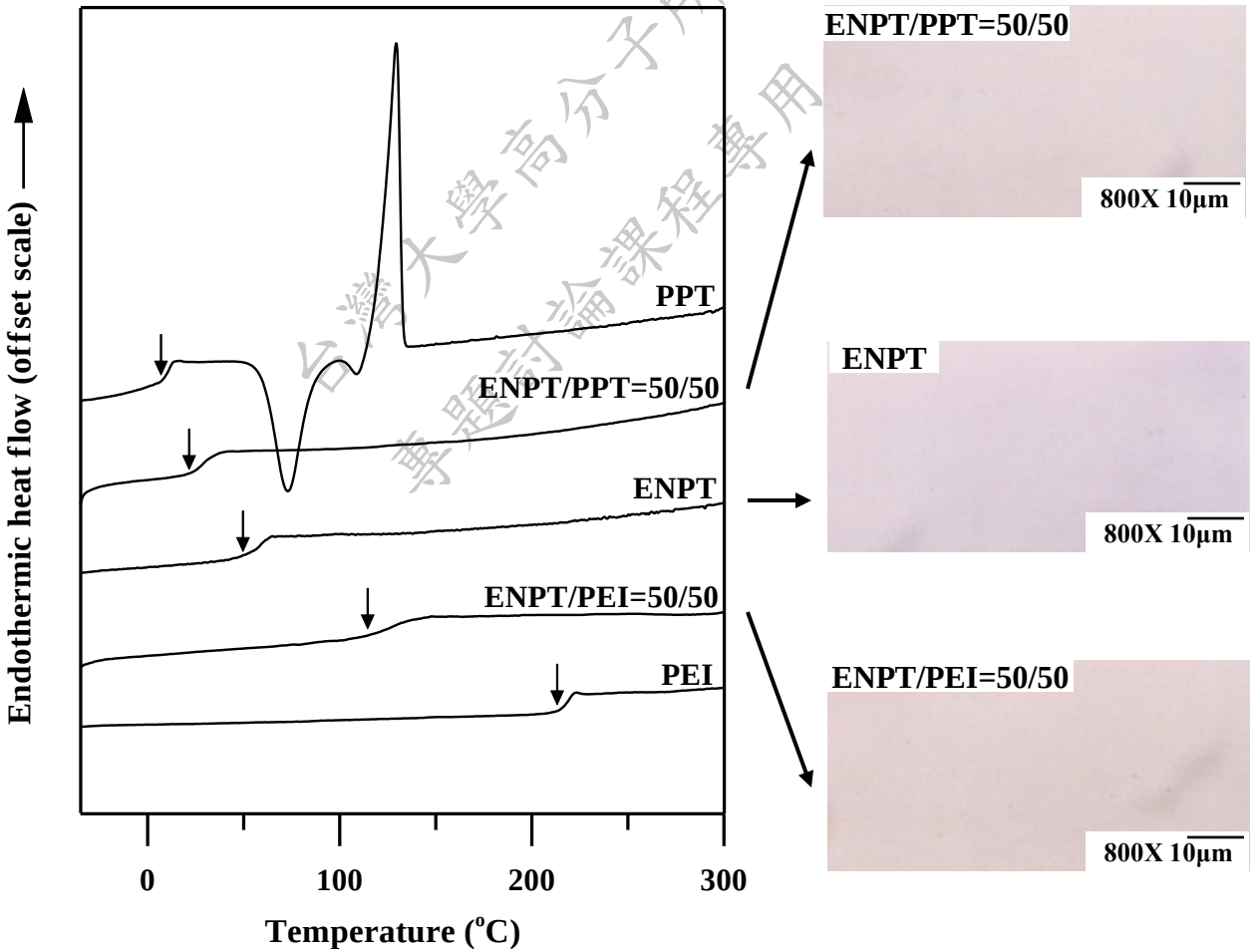
ENPT/PEI binary blends

❖ **DSC & POM results of ENPT blends**



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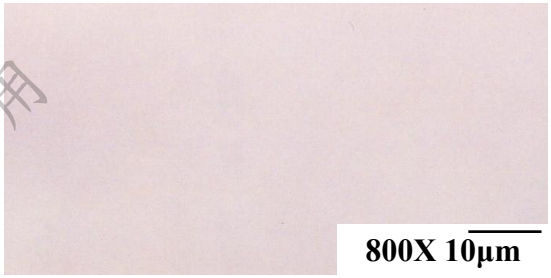
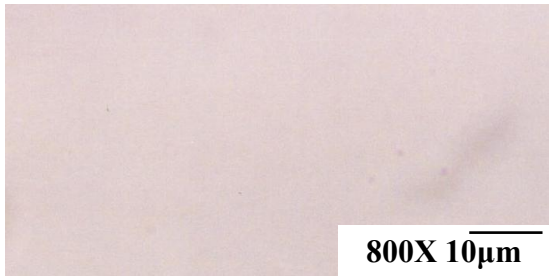
ENPT/PEI binary blends

❖ **POM graphs of ENPT/PEI blends**

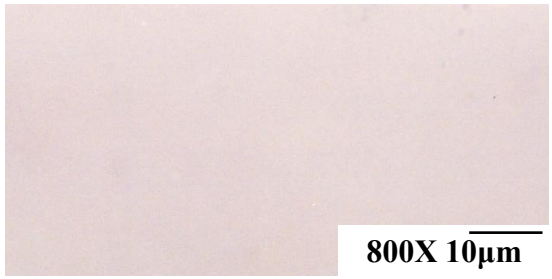
80/20



66/33



50/50



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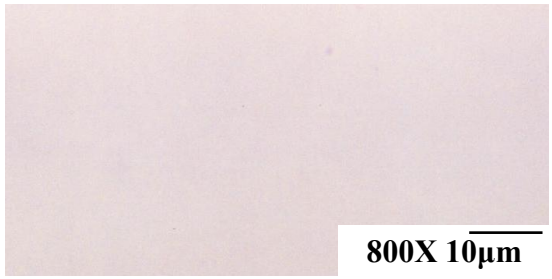

20/80

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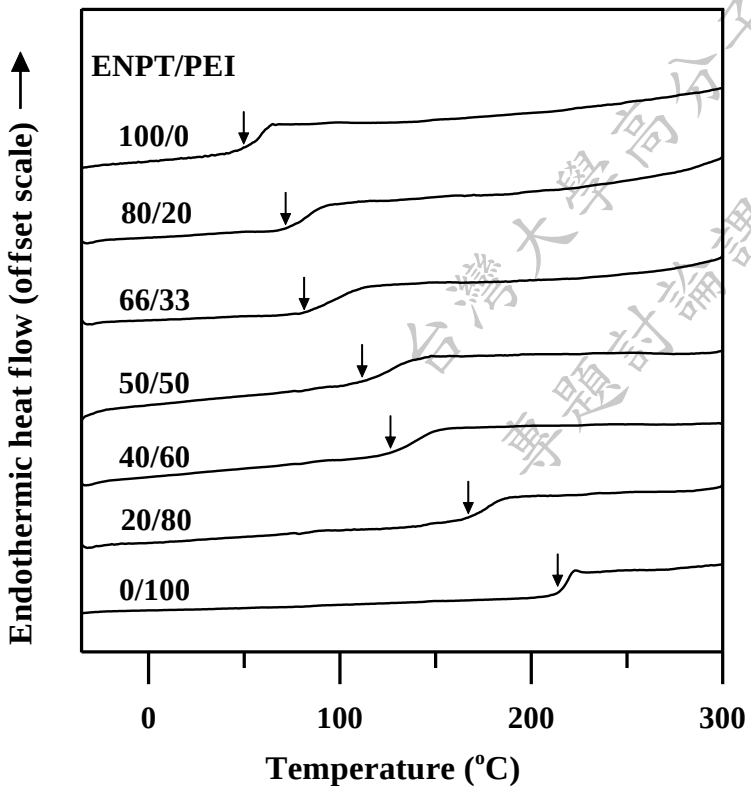
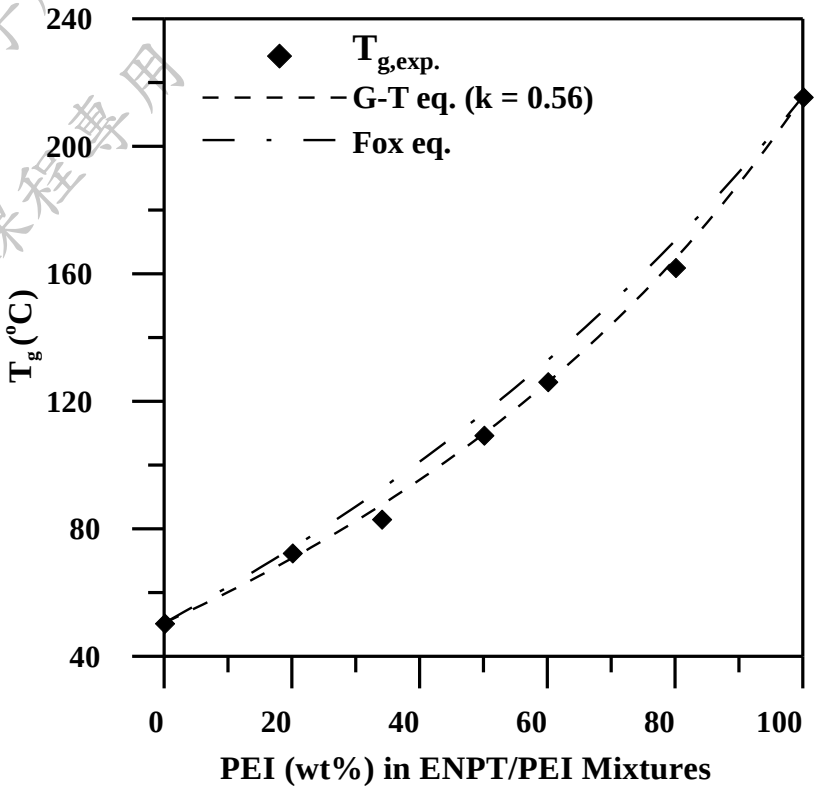
專題討論課程專用

ENPT/PEI binary blends

❖ DSC & T_g fitting of ENPT/PEI blends



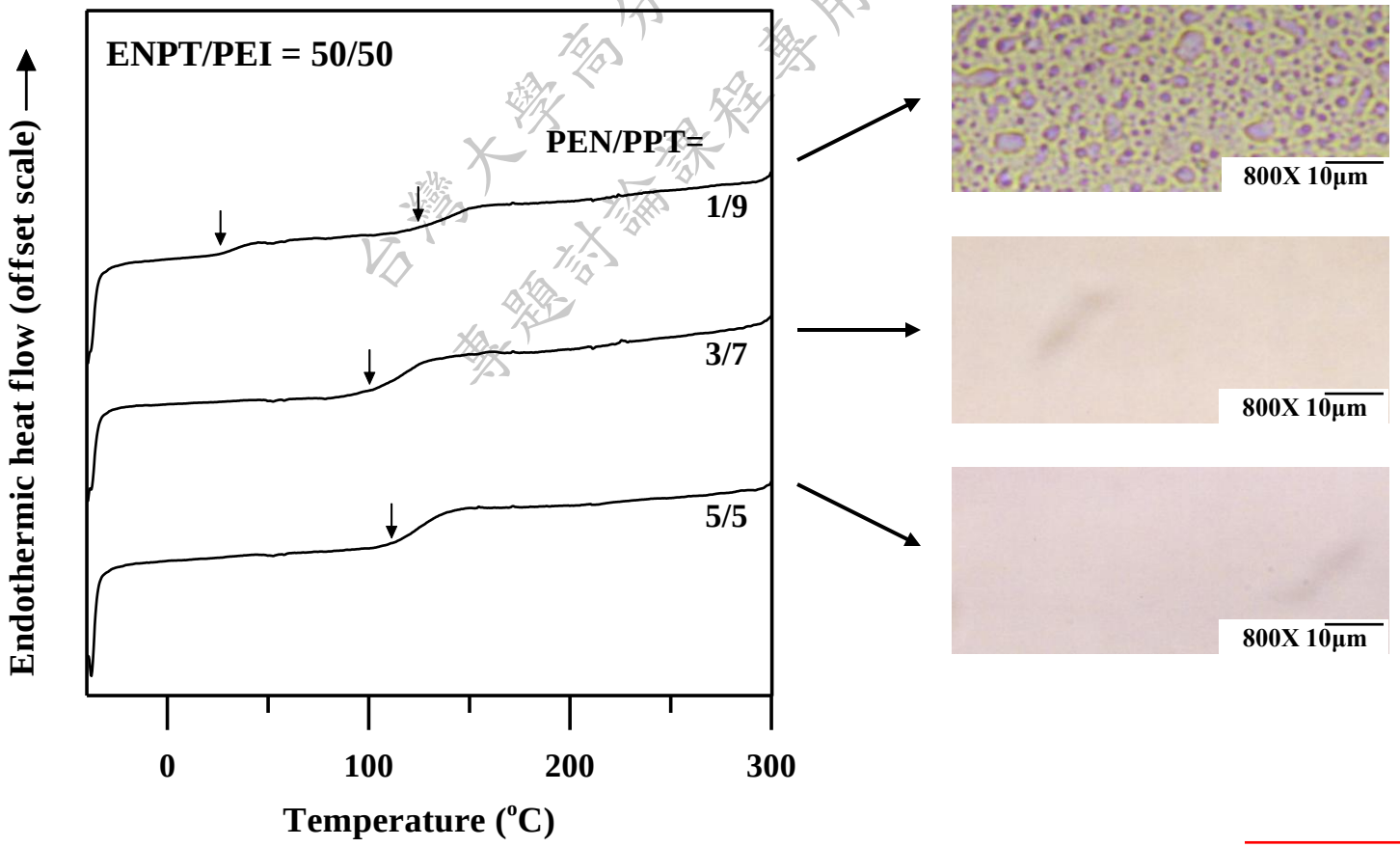


台灣大學高分子所
專題討論課程專用

Effects of PEN/PPT ratio

❖ **The effect of different compositions on miscibility**

ENPT is composed of different PEN/PPT ratios : (A) 1/9, (B) 3/7, (C) 5/5. (w/w)



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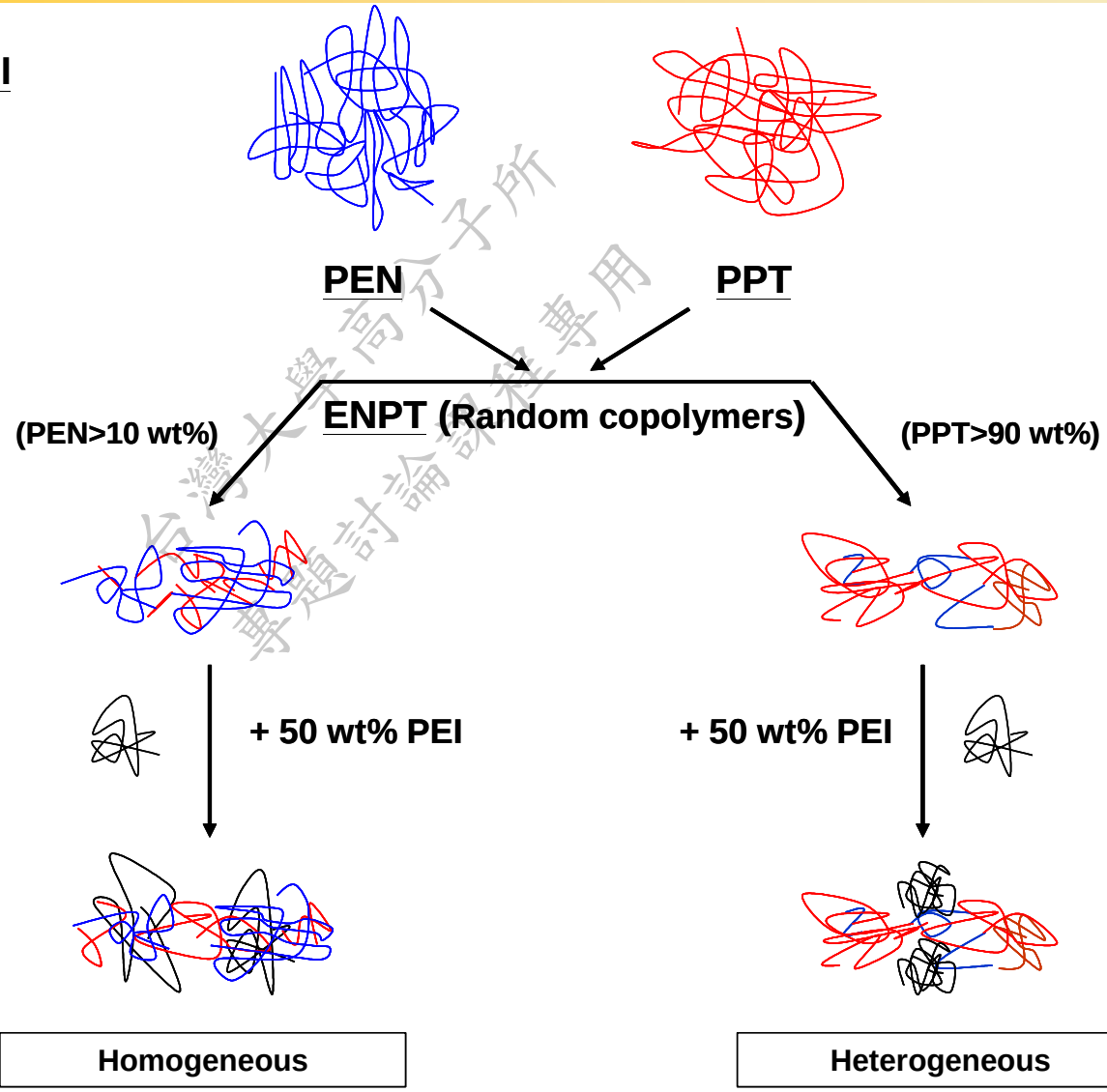
PEN/PPT/PEI



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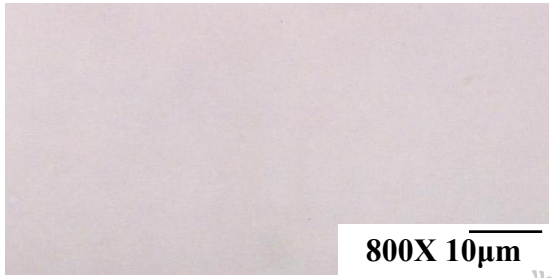
ENTT/PEI binary blends

❖ **POM graphs of ENTT/PEI blends**



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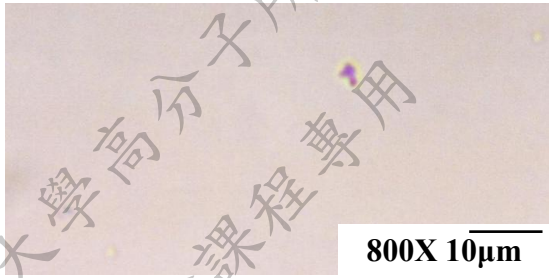
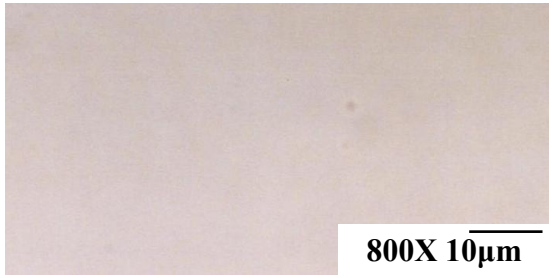
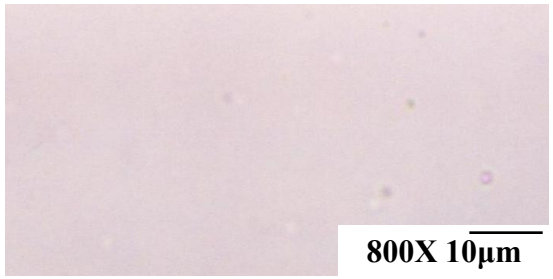


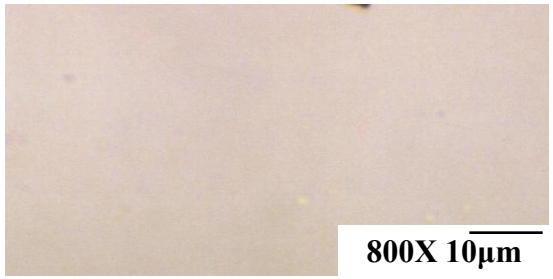
80/20



66/33



50/50



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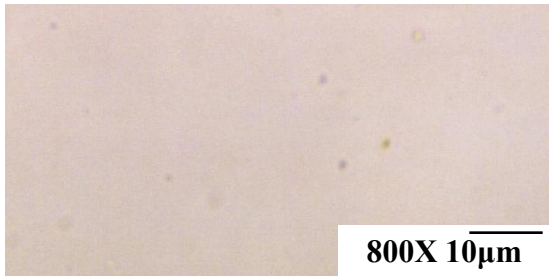

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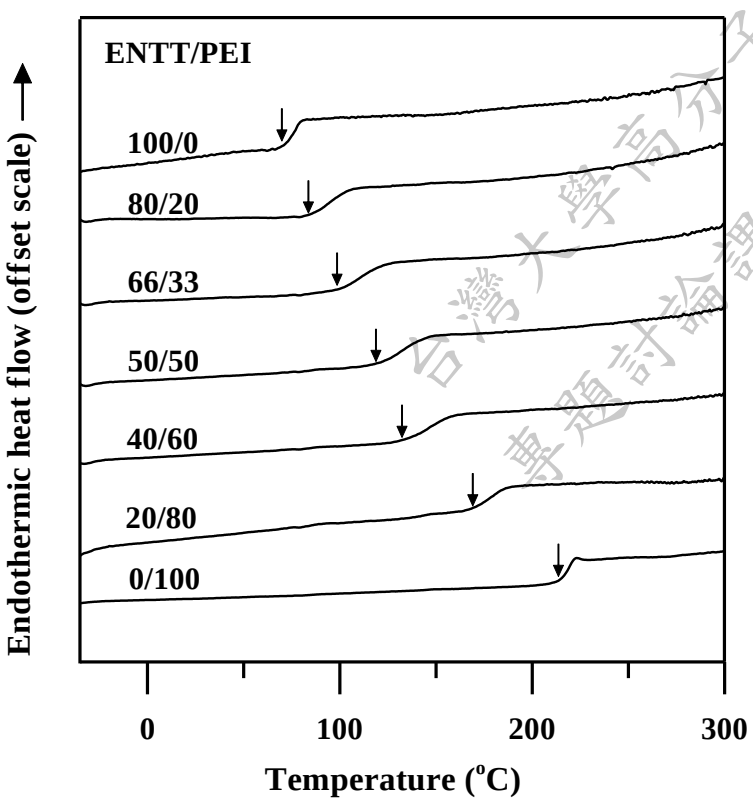
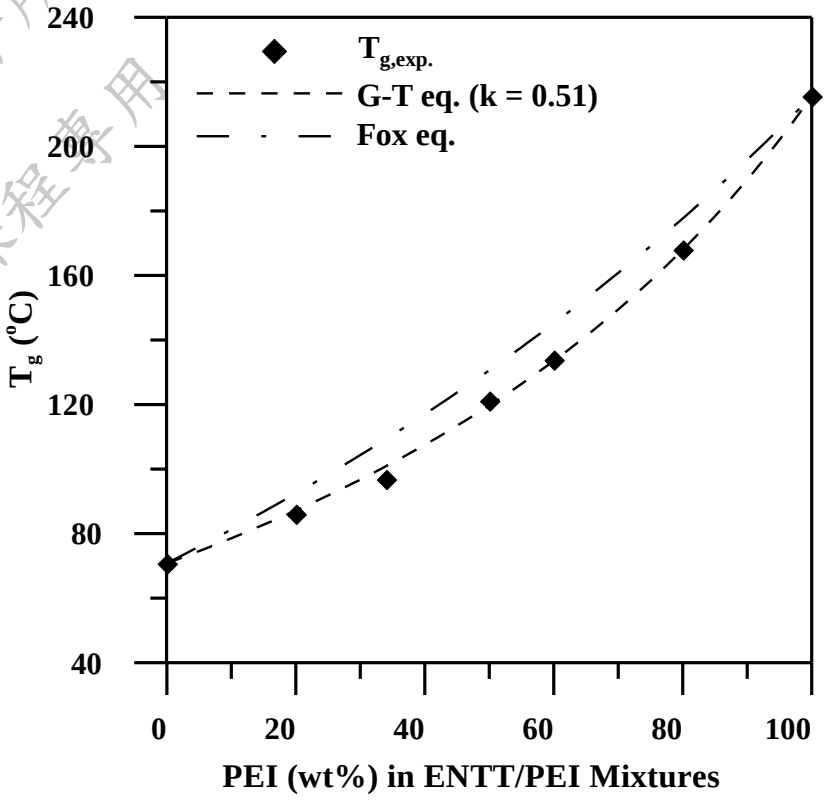
專題討論課程專用

ENTT/PEI binary blends

❖ DSC & T_g fitting of ENTT/PEI blends





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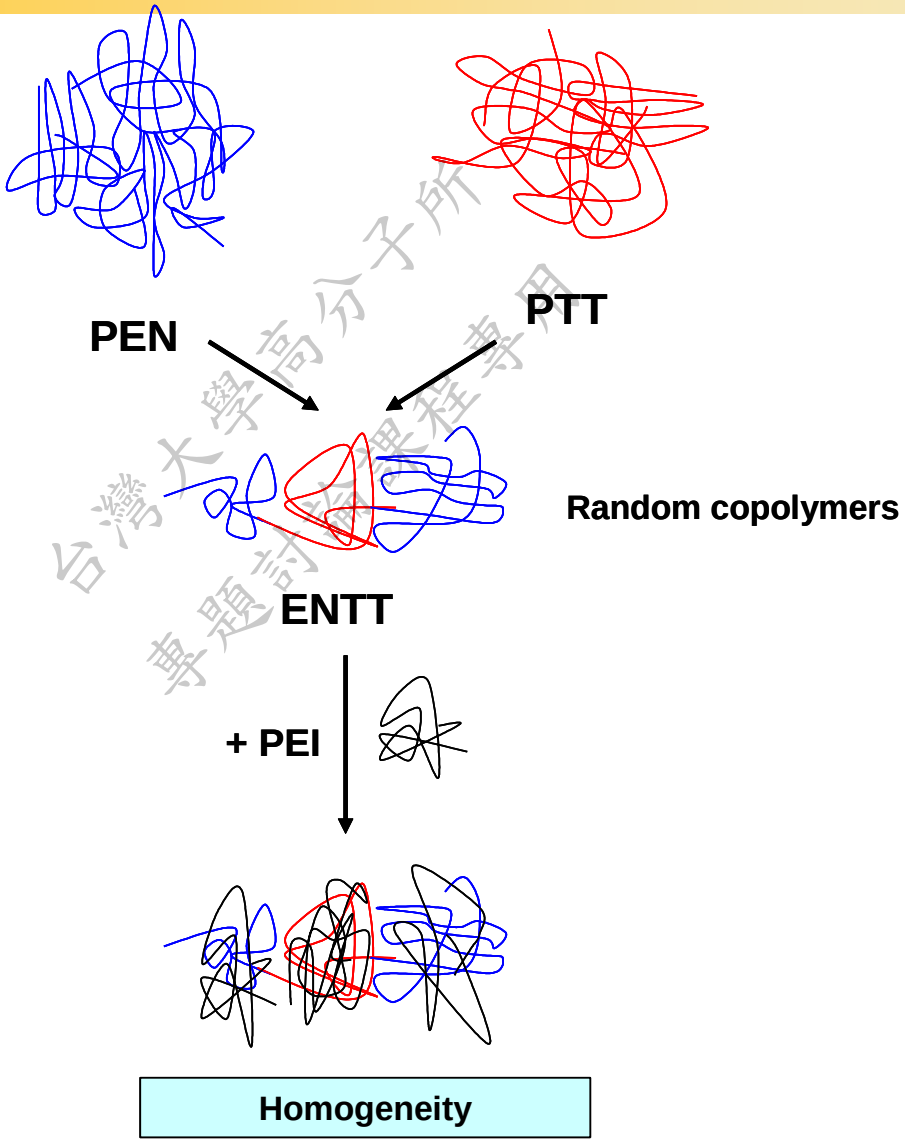
PEN/PTT/PEI



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ENTT/PEI binary blends

❖ T_g comparisons between binary and ternary blends

Glass transition temperature (°C)				
PEI wt%	PEN/PPT/PEI (300°C 60min)	ENPT/PEI	PEN/PTT/PEI (300°C 30min)	ENTT/PEI
0	50	50.5	72.5	70.8
20	71.3	72.6	85.9	86.1
33	81	83.2	97	96.9
50	105.4	109.5	117.5	121.2
60	125.8	126.3	136.5	133.9
80	161.9	162.1	167	168.5
100	215.6	215.6	215.6	215.6

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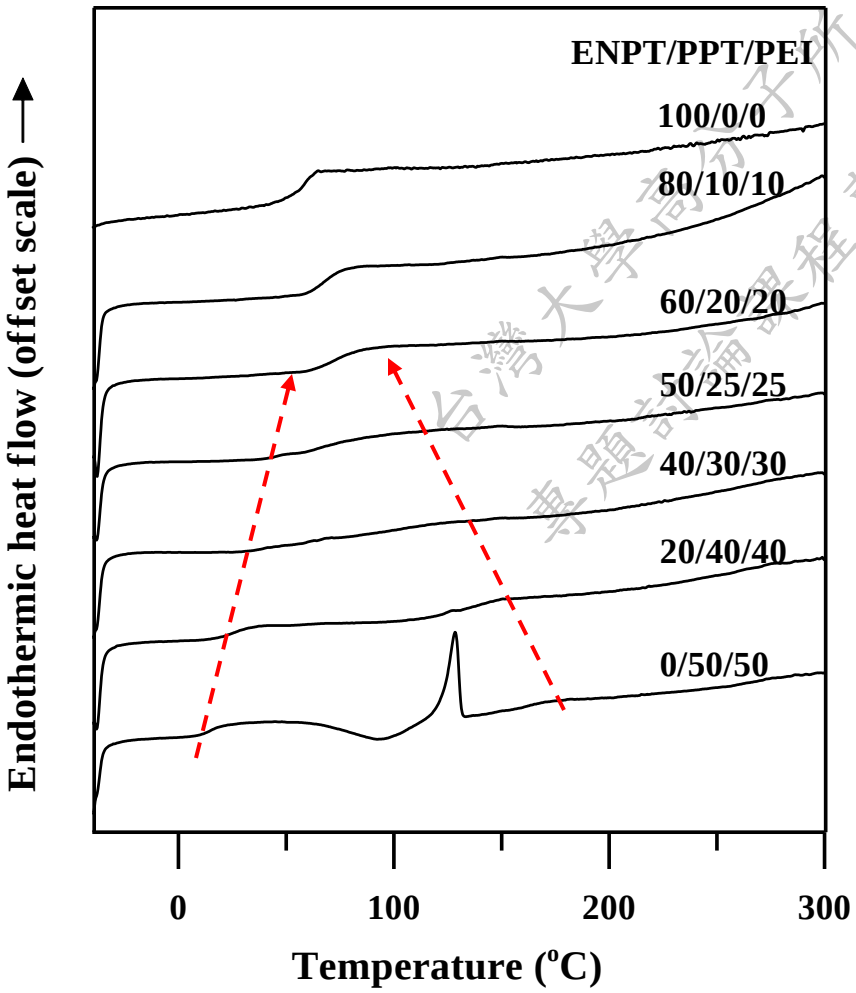
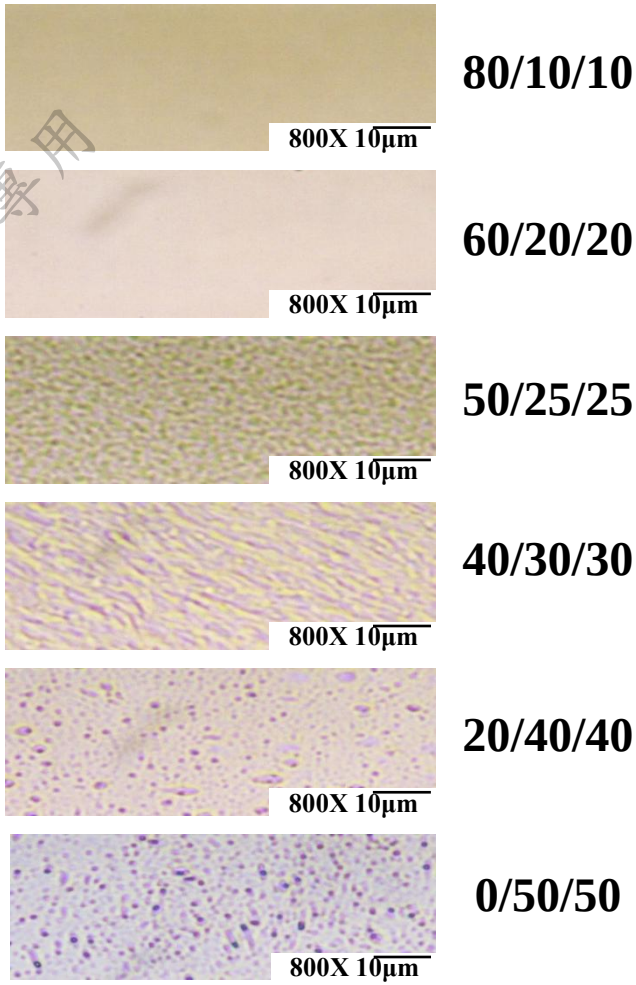
ENPT/PPT/PEI ternary blends

❖ **DSC & POM results**





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Conclusion

- **PPT/PEI and PHT/PEI blednds**

methylene moieties in the repeating unit of the aryl polyester ↑ → **the interactions between the aryl polyester and PEI** ↓

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PEN/PPT/PEI and PEN/PTT/PEI ternary blends

The transesterification in PEN and aryl polyesters enhanced the miscibility of the PEN/Aryl polyesters/PEI blends

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ENPT and ENTT copolymers

Reactively Formed ENPT Copolymers as Compatibilizers in Ternary Blends of PEN/Aryl Polyesters /PEI

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Green Materials and Resources Lab.


The End

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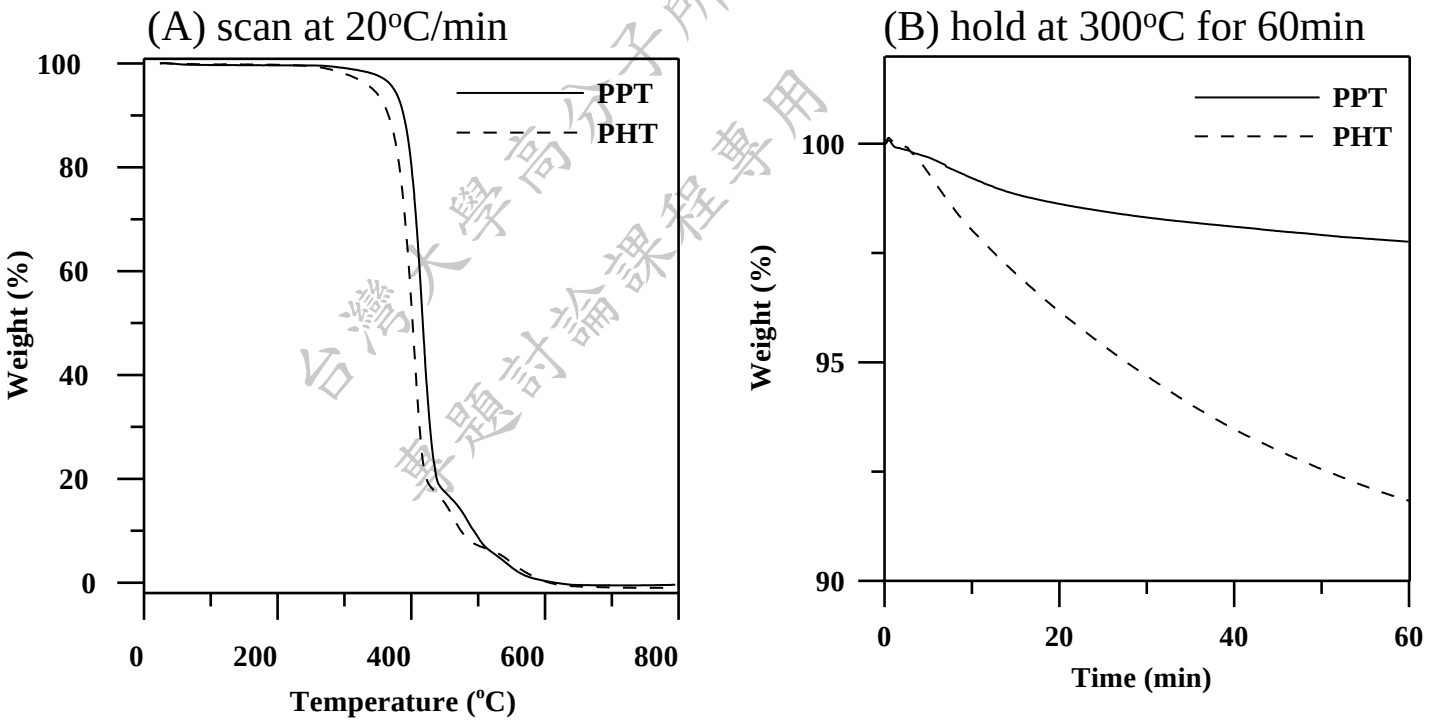
TGA tests

❖ **TGA tests of PPT and PHT**



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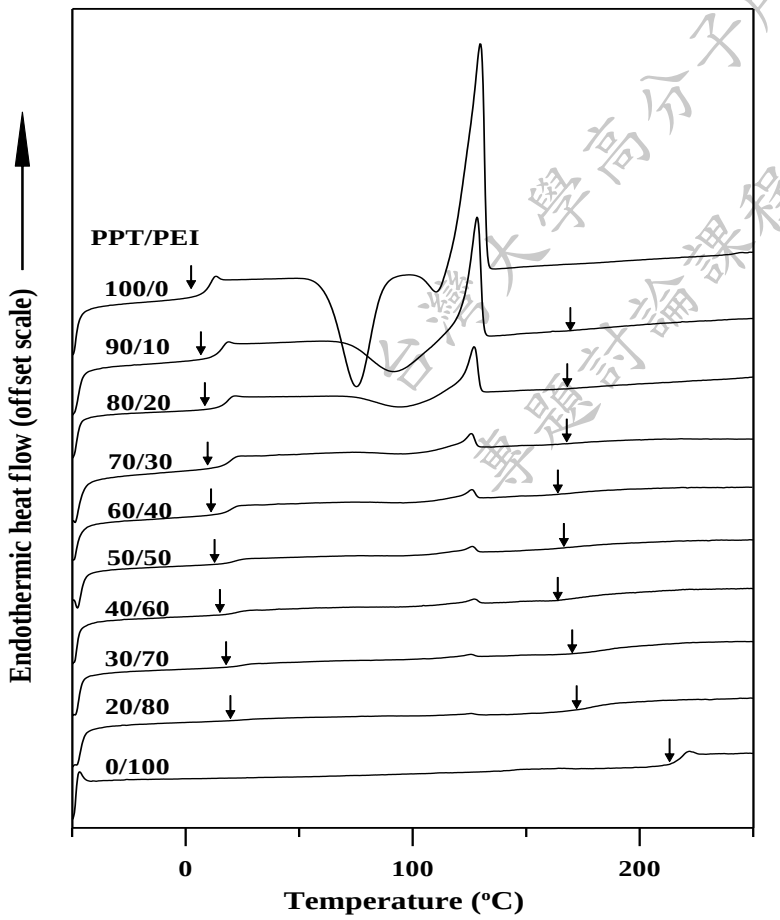
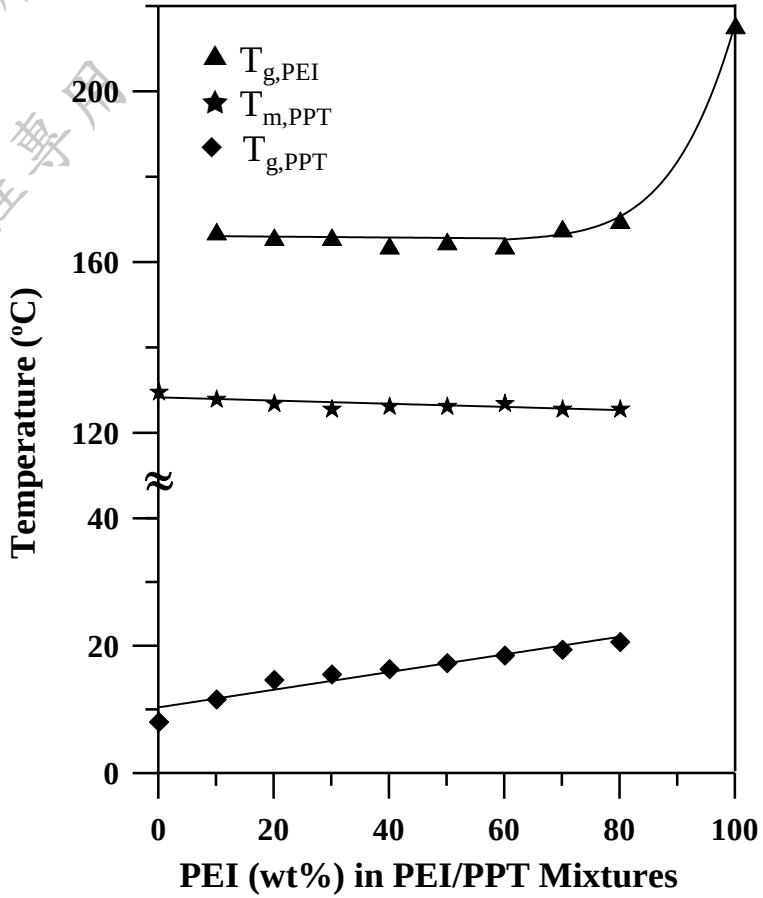
PPT/PEI : Melt-blending & Co-precipitation
PHT/PEI: Co-precipitation

PPT/PEI binary blends

❖ **DSC traces of melt-blended PPT/PEI blends**





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Conclusion

- **PPT/PEI及PHT/PEI兩成分摻合系統：**

  經POM觀察相形態及DSC分析T_g，證實此兩成分摻合系統為不相容，經計算得分子間作用力參數(χ_{12})：PPT/PEI之χ_{12}=0.12±0.01、PHT/PEI之χ_{12}=0.17±0.01。進一步發現主鏈上碳數增加使aryl polyester的分子鏈運動性增加而羰基(carbonyl)密度降低，導致與PEI的作用力減弱，系統由相容轉變為相分離。

- **PEN/PPT/PEI與PEN/PTT/PEI三成分摻合系統：**

  實驗發現PEN/PPT/PEI摻合為不相容之系統，而PEN/PTT/PEI摻合為部分相容；但兩系統經高溫熱處理則所有組成變為均相。以^{1}H-NMR分析證實交酯化反應發生於PEN/PPT及PEN/PTT間並產生共聚合物。由玻璃轉化行為、溶解度及結晶行為，可進一步觀察到高分子鏈結構隨反應時間改變的情形。

- **ENPT/PEI及ENTT/PEI摻合系統:**

  ENPT/PEI為相容之摻合系統，說明當PEN含量超過30wt%時，PEN與PPT交酯化提升PPT與PEI之親和性而使PPT/PEI相容。實驗也發現ENPT對PPT/PEI摻合具有助容劑的效果。此外，ENTT/PEI也為一相容的摻合系統。